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ELASTICITY CHARACTERISTICS OF MATERIALS AT HIGH TEMPERATURE, (U)
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ELASTICITY CHARACTERISTICS OF MATERIALS
AT HIGH TEMPERATURE

by

Yu. A. Kashtalyan

FOREIGN TECHNOLOGY DIVISION

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ELASTICITY CHARACTERISTICS OF MATERIALS AT HIGH TEMPERATURE

By: Yu. A. Kashtalyan

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U. S. BOARD ON GEOGRAPHIC NAMES TRANSLITERATION SYSTEM

Block	Italic	Transliteration	Block	Italic	Transliteration
А а	*А а*	A, a	Р р	*Р р*	R, r
Б б	*Б б*	B, b	С с	*С с*	S, s
В в	*В в*	V, v	Т т	*Т т*	T, t
Г г	*Г г*	G, g	У у	*У у*	U, u
Д д	*Д д*	D, d	Ф ф	*Ф ф*	F, f
Е е	*Е е*	Ye, ye; E, e*	Х х	*Х х*	Kh, kh
Ж ж	*Ж ж*	Zh, zh	Ц ц	*Ц ц*	Ts, ts
З з	*З з*	Z, z	Ч ч	*Ч ч*	Ch, ch
И и	*И и*	I, i	Ш ш	*Ш ш*	Sh, sh
Й й	*Й й*	Y, y	Щ щ	*Щ щ*	Shch, shch
К к	*К к*	K, k	Ъ ъ	*Ъ ъ*	"
Л л	*Л л*	L, l	Ы ы	*Ы ы*	Y, y
М м	*М м*	M, m	Ь ь	*Ь ь*	'
Н н	*Н н*	N, n	Э э	*Э э*	E, e
О о	*О о*	O, o	Ю ю	*Ю ю*	Yu, yu
П п	*П п*	P, p	Я я	*Я я*	Ya, ya

*ye initially, after vowels, and after ъ, ь; e elsewhere. When written as ё in Russian, transliterate as yё or ё.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	$\sinh^{-1}$
cos	cos	ch	cosh	arc ch	$\cosh^{-1}$
tg	tan	th	tanh	arc th	$\tanh^{-1}$
ctg	cot	cth	coth	arc cth	$\coth^{-1}$
sec	sec	sch	sech	arc sch	sech^{-1}
cosec	csc	csch	csch	arc csch	csch^{-1}

Russian	English
rot	curl
lg	log

ELASTICITY CHARACTERISTICS OF MATERIALS AT HIGH TEMPERATURE.

Yu. A. Kashtalyan.

In the book are described the experimental procedures of determining elasticity characteristics (Young's modulus, shear modulus, Poisson ratio) of materials at high temperatures.

Are examined construction/designs and operating principle of devices which make it possible to measure the characteristics indicated to temperature of 3000° K. Are given the new data on elasticity characteristics of refractory metals (W, Mo, Nb, Ta), of alloys on their basis, different refractory compounds, and also Pyrocerams and ferroglass materials at normal and high temperatures.

It is designed for scientific and technical-engineering workers, instructors, graduate students and students of VUZ [Institute of Higher Education].

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PREFACE.

In the development of the majority of the branches of new technology, considerable role belongs to the materials which can reliably function at high temperatures. Such materials are especially necessary for the creation of the new types of nuclear reactors, of rocket engines, generators of direct transformation of thermal energy into electrical, the settings up of chemical industry and metallurgical equipment. Operating temperatures, which reach in some of the mentioned devices 3000-3500° K, subsequently will be raised, for which will be required the development of the new types of materials.

Since the conditions of work at the high temperatures in each branch of technology considerably differ, to high-temperature materials are presented the most diverse requirements. These requirements they can satisfy metallic and nonmetallic materials. The correct selection of material for the appropriate conditions of work can be made only in such a case, when will be accurately known its physicomechanical properties over a wide range of temperatures. The need for the definition of the values, which characterize these

properties, appears both during the development of new high-melting materials and during an improvement in the qualities already known.

A number of most important physicomechanical properties includes the elasticity of material, which determines in essence according to the values of Young's modulus, modulus of shear and Poisson ratio. These values, which obtained also the name of elasticity characteristics, are utilized during theoretical and experimental studies and enter in all calculations for strength, rigidity and heat resistance of the elements of construction/designs. If are known the values of elasticity characteristics of material, then on them can be designed and its other physical characteristics: the characteristic temperature, root-mean-square atomic displacement in the units of crystal lattice, etc.

The values of the moduli of elasticity of high-melting materials at temperatures to 2500-3000° K, which for some materials are workers, they were not known, since were not developed the corresponding methods and settings up for their determination.

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This monograph is dedicated to the analysis of elasticity characteristics of high-melting materials at high temperatures. In it

are briefly presented the basic information about elasticity characteristics of the material: Young's modulus, modulus of shear and Poisson ratio are examined the existing methods of their determination at high temperatures. Are given the detailed descriptions of the developed in Institute problems of the strength of AS UkSSR of special procedures and corresponding settings up for measuring Young's modulus and shift/shear of materials in the range from room temperature to 3000° K. The data, obtained during these settings up, give the possibility calculation to determine the value of Poisson ratio. Together with the description of procedures and settings up, in monograph are given the experimental data on elasticity characteristics at the high temperatures of the refractory metals of "large tetrad" - tungsten, molybdenum, niobium, tantalum, their alloys and a series of refractory compounds, and also of some pyrocerams, glasses and metal-glass materials.

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Chapter I.

GENERAL INFORMATION ABOUT ELASTICITY CHARACTERISTICS OF HIGH-MELTING MATERIALS.

For manufacturing the parts of different machines and apparatuses, that work at high temperatures, are utilized the refractory metals and alloys, refractory compounds, graphite, vitreo-crystalline and metal-glass materials. The materials indicated in the majority of the cases are polycrystalline ones and their elastic behavior is determined by distance/removal, approach or shift/shear of atoms. Although the elastic properties of separate crystal depend on the direction of crystal faces, in the polycrystalline body, which consists of the disorderly arranged/located crystallites, they do not depend on direction. Therefore polycrystalline bodies it is accepted to consider isotropic.

The elastic behavior of any isotropic body is characterized by elastic modulus E (Young's modulus), by modulus of shear G, by bulk modulus K (bulk modulus) and by Poisson ratio μ. From elementary

Hooke's law for strains in elastic region, it follows that values E, G, K show proportionality between voltages and tensile strain, displacement and cubic compression:

$$\sigma = E\varepsilon; \quad (1)$$
$$\tau = G\gamma; \quad (2)$$
$$p' = K\frac{\Delta V}{V}, \quad (3)$$

while coefficient μ characterizes a change in the volume of body during strain. These values are connected by the relationship/ratios

$$\mu = \frac{E}{2G} - 1; \quad (4)$$
$$K = \frac{E}{3(1-2\mu)}. \quad (5)$$

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Characterizing the elastic behavior of polycrystalline body, the moduli of elasticity thereby determine the strength of interatomic communication/connections in crystal lattice; therefore in terms of their values can be estimated and other physical quantities: heat of sublimation, melting point, enthalpy, energy of the activation of diffusion and self-diffusion, standard deviation of atoms from position of equilibrium, characteristic temperature, coefficient of linear expansion, etc. For some of these values, are establish/installed the numerical ratios, therefore, they can be calculated in the value of the modulus of elasticity, and vice versa. This can prove to be useful, when to directly measure the modulus of

elasticity is difficult, for example, at the very high temperatures, close to melting points.

Between the coefficient of linear expansion and the modulus of elasticity is established following dependence [65]:

$$\alpha \approx \frac{k}{R_0^3 E}, \qquad (6)$$

where R_0 - equilibrium interatomic distance; k - Boltzmann constant.

Especially frequently utilize values of the moduli of elasticity for calculating such values as characteristic Debye temperature [64]:

$$\Theta' \approx \frac{1{,}68 \cdot 10 \sqrt[3]{E}}{A^{\frac{1}{3}} d^{\frac{1}{6}}} \qquad (7)$$

(d - density, A - atomic weight) and the standard deviation of atoms from position of equilibrium:

$$\overline{U}^2 = \frac{1{,}49 \cdot 10^{-23} d^{\frac{1}{3}} (T')^2}{E^{\frac{3}{2}}} \quad \text{for} \quad T' < \frac{\Theta'}{8}; \qquad (8)$$

$$\overline{U}^2 = \frac{1{,}52 \cdot 10^{-20} T'}{V^{\frac{1}{3}} E} \quad \text{for} \quad T' > 1{,}6\,\Theta' \qquad (9)$$

(V - atomic volume, Å³).

For intermediate temperatures $\theta/8 < T < 1.6\theta'$ value $\bar{U}^2$ can be determined from Debye- Veler's precise relaticnship/ratio.

The module/moduli cf elasticity as other physical quantities which are determined by the strength of interatcmic communication/connectiors, depend on the positicn of cell/element in periodic system.

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Cn Fig. 1 and 2 shown alterraticn in the mcdule/modulus of normal elasticity and shear modulus in dependence cn the atomic number of cell/element [52]. There is no complete agreement here all the same, since the maximum value cf Ycung's modulus is observed not in the tungsten, which has the highest melting point, but in osmium. The high values of the moduli of elasticity have transition metals, especially those whose melting pcint reaches 3000° K and are above - osmium, rhenium, tungsten.

Since the moduli of elasticity of material reflect the strength of its interatomic communication/connections, in perfect crystal they could serve as the indices cf its strength - the stronger interatomic

communication/connections, by the fact more high voltage must be applied in order to break them. The real strength of crystals and polycrystalline materials is much lower than the theoretical, determined by interatomic communication/connections, which is explained by the presence of a large quantity of flaw/defects (dislocation, grain boundary and interstitial atoms). Effect of these flaw/defects on the elastic properties of materials not as considerable as on plastic ones. For real materials there are no precise relationship/ratios between module/moduli of elasticity, ultimate strength and hardness, although certain correlation is observed.

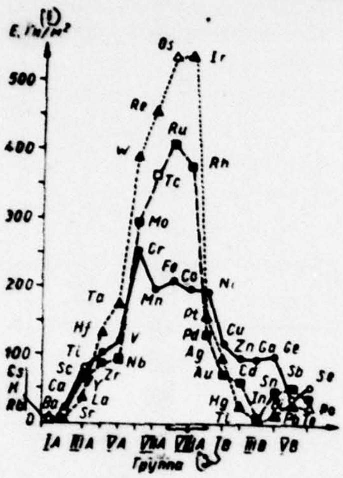

Fig. 1.

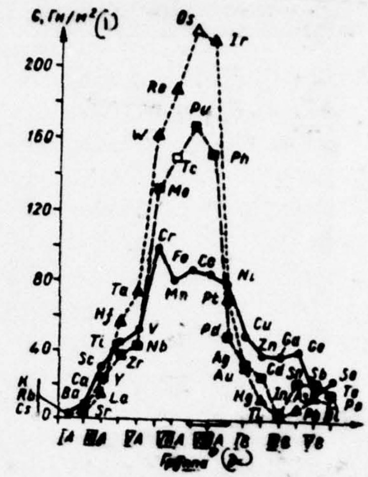

Fig. 2.

Fig. 1. Change of Young's modulus of transition metals in dependence on their position in periodic system.

Key: (1). H/m². (2). Group.

Fig. 2. Change of modulus of shear of transition metals in dependence on their position in periodic system.

Key: (1). H/m². (2). Group.

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The modulus of elasticity, similar to other physical quantities,

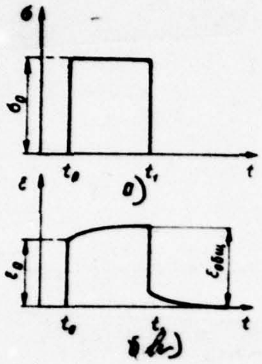
is changed their value under the influence of different fields on solid (mechanical, temperature, magnetic, electrical, etc.), if this effect affects binding forces between atoms.

Temperature exerts the strongest influence on the moduli of elasticity, since its increase causes an increase in the fluctuations of atoms around position of equilibrium and an increase in the distance between them. It is considered that with an increase in the temperature by one degree the value of module/moduli is decreased by 0.03o/c [36].

During the evaluation of the effect of the field of mechanical forces, it is necessary to take into consideration, what expressions (1)-(3) describe the process of deformation of ideal elastic body. The mechanical analog of this body can be represented in the form of spring. Real solids with low voltages develop inelasticity, i.e., during their deformation is observed the delay of strain on phase from voltage. Graphically this is shown on Fig. 3.

The delay of strain from the voltage in real solid is caused by the processes which can have different nature. So, if solid is deformed under adiabatic conditions, its temperature slightly changes: with elongation it is depressed, and during compression it grew/rises (Fig. 4). During the instantaneous application/appendix of

tensile stress σ, the specimen/sample is deformed to value ε_0 and its temperature is depressed. At this moment its modulus of elasticity is defined by the tangent of angle α and is ne-relaxed, or as it still are called, adiabatic. In certain time the temperature of specimen/sample will be equaled with the temperature of surrounding medium and specimen/sample will be lengthened to value AB.



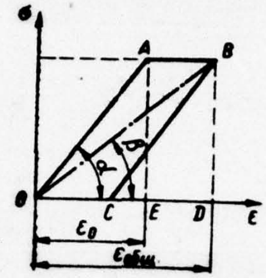


Fig. 3.

Fig. 4.

Fig. 3. Dependence of stress (a) and of strain (b) from time for relaxation process.

Fig. 4. Schematic of thermal relaxation.

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Now its modulus of elasticity, determined by the tangent of angle β, will be that relaxed, or "by isothermal", by module/modulus.

The differencee between these module/moduli small and is determined from the expression

$$\frac{E_{\text{из}}}{E_{\text{ад}}}=1-\frac{\alpha T E_{\text{из}}}{\rho C_p}, \qquad (10)$$

where α - a coefficient of linear expansion; T - absolute

temperature; ρ - specimen/sample density; C_p - specific heat capacity at constant pressure.

For describing the inelastic behavior of metals, most frequently is utilized the mechanical analog solid K. Zener [18], the so-called standard linear solid (Fig. 5). Stress and strain of this body are connected by the relationship/ratio

$$\sigma + \tau_\varepsilon \dot{\sigma} = M_p (\varepsilon + \tau_\sigma \dot{\varepsilon}), \quad (11)$$

where τ_ε - time of relaxation of stress under the condition of constant strain; τ_σ - time of retardation, i.e., the value, which characterizes the rate of an increase in the strain with constant stress; M_p - relaxed modulus of elasticity.

The module/modulus which establishes communication/connection between the stress and the strain under conditions when relaxation does not manage to occur, i.e., nonrelaxed, it is designated through $M_н$. Value

$$\Delta M = M_н - M_p \quad (12)$$

is called the flaw/defect of module/modulus. The degree of relaxation of module/modulus is determined by the expression

$$\Delta = \frac{M_н - M_p}{M_н}. \quad (13)$$

Sometimes this value is alsc called the flaw/defect of module/modulus.

During repeated deformation the module/modulus will be unrelaxed in such a case, when the time between separate cycles is small so that would occur the additicnal strain. Relaxed module/modulus will be when the time between separate cycles is greater than relaxation time.

It is important to establish/install communication/connection between the stress and the strain in the standard linear body when these values periodically change in time. For this, the stress and strain represent as the periodic functions cf time and substitute in equaticn (11).

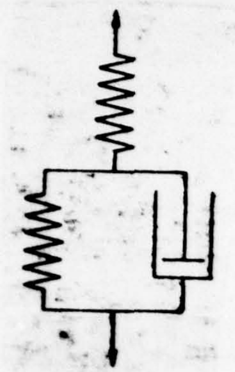

Fig. 5. The mechanical analog of standard linear solid.

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For fluctuations with angular frequency ω, we have

$$\sigma(t) = \sigma e^{i\omega t};\ \varepsilon(t) = \varepsilon e^{i\omega t}. \qquad (14)$$

After the substitution of expression (14) into equation (11) we obtain

$$\sigma(1 + i\omega\tau_\varepsilon) = M_p \varepsilon(1 + i\omega\tau_\sigma). \qquad (15)$$

From equation (15) it follows that between the relaxed and ne-relaxed module/modulus there is the dependence

$$M_n = \frac{\sigma(\omega \to \infty)}{\varepsilon(\omega \to \infty)} = \frac{\tau_\sigma}{\tau_\varepsilon} M_p. \qquad (16)$$

Communication/connection between the stress and the strain is

establish/installed by equation (15) means of the module/modulus, which represents the complex quantity:

$$\sigma = \frac{1 + i\omega\tau_\sigma}{1 + i\omega\tau_\epsilon} M_p \epsilon. \qquad (17)$$

Value

$$M^* = \frac{1 + i\omega\tau_\sigma}{1 + i\omega\tau_\epsilon} M_p \qquad (18)$$

is called the complex modulus of elasticity. During repeated deformation this module/modulus determines not only absolute the value of strain, but also its displacement in time with respect to stress. The phase angle between the stress and the strain is determined by the dissipation of energy during fluctuations. The tangent of this angle is equal to the ratio/relation of the imaginary and real parts of the complex module/modulus

$$\operatorname{tg}\varphi = \frac{I(M^*)}{R(M^*)} = \omega \frac{\tau_\sigma - \tau_\epsilon}{1 + \omega^2 \tau_\sigma \tau_\epsilon}. \qquad (19)$$

If we introduce the geometric mean of two values of relaxation time $\tau = \sqrt{\tau_\epsilon \tau_\sigma}$ and geometric mean of two module/moduli $\overline{M} = \sqrt{M_H M_P}$ then

$$\operatorname{tg}\varphi = \frac{\Delta M}{\overline{M}} \cdot \frac{\omega\tau}{1 + \omega^2\tau^2}. \qquad (20)$$

As the measure of the ratio of stress to strain, are accepted the absolute value of the complex module/modulus M^* or its real part $R(M^*)$.

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More frequently is utilized the latter and is defined dynamic modulus as causing that part of the strain which is located in phase with the stress:

$$M_{\text{д}} = M_{\text{р}} \frac{1 + \omega^2 \tau_\sigma^2}{1 + \omega^2 \tau_\sigma \tau_\varepsilon}, \tag{21}$$

or

$$M_{\text{д}} = M_{\text{н}} \left(1 - \Delta \frac{1}{1 + \omega^2 \tau^2}\right). \tag{22}$$

Expressions for dynamic modulus (22) and tangent of the phase angle between the stress and strain (20) are the symmetrical functions of product $\omega\tau$. Analyzing these expressions, we find: at the high frequencies when $\omega \to \infty$, $M_{\text{д}} \to M_{\text{н}}$, tg $\varphi \to 0$; at the low frequencies when $\omega \to 0$, $M_{\text{д}} \to M_{\text{р}}$, tg $\varphi \to 0$; when $\omega\tau = 1$, when the dissipation of energy is maximum, $M_{\text{д}} = \frac{M_{\text{н}} + M_{\text{р}}}{2}$. A change in values tg φ and $M_{\text{д}}$ in dependence on value $\omega\tau$ is shown on Fig. 6.

The dissipation of energy is caused by different processes, which occur within solid, each process having only in it the which is inherent relaxation time. This leads to the fact that during a change in the frequency ω the dissipation of energy on separate frequencies reaches maximum values. The dependence of the dissipation of energy on the frequency of loading was called relaxation spectrum (Fig. 7).

With an increase in the frequency, the value of the peaks of the spectrum is decreased, therefore, the relaxation of the modulus of elasticity will be less.

The relaxation time of the processes, connected with the displacement of atoms, depends on the temperature:

$$\tau=\tau_0 e^{\frac{H}{RT}}, \qquad (23)$$

where H - an energy of the activation of this relaxation process; R - universal gas constant; T - absolute temperature.

According to formula (23) the temperature has strong effect on relaxation time: with increase its relaxation time is decreased and the peaks of relaxation spectrum are misaligned into the region of more high frequencies.

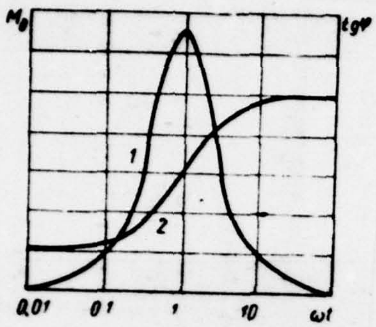

Fig. 6. Dependence of the dispersion/dissipation of energy (1) and of dynamic modulus (2) on frequency.

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Consequently in order to obtain its values of dynamic modulus at high temperatures, close to that ne-relaxed, measurement under these conditions it should be carried out at more possible high frequencies.

As an example Fig. 8 gives the curves of the dependences of the modulus of shear of single-crystal and polycrystalline aluminum on the temperature, obtained at frequency 0.8 Hz [18]. Relaxation of the modulus of shear of polycrystalline specimen/sample, occurring at temperatures are higher than 470° K and are achieved several ten percent, it is caused by viscous flow on grain boundaries.

It should be noted that at the high temperatures in material can occur the irreversible processes, which affect the ne-relaxed module/modulus and the flaw/defect of module/modulus. This always must be born in mind during the analysis of the temperature dependences of the moduli of elasticity.

There is large interest in the explanation of a question concerning effect composition and porosity on the moduli of elasticity of real materials. The foreign atoms which can hit crystal lattice, they change strength of interatomic communication/connections, which causes an increase or the decrease of module/moduli. However, constant additions do not have great effect on the moduli of elasticity, since even with their considerable quantity the absolute values of module/moduli change in all on several percentages. So, Young's modulus the majority of steels, which have different composition, is equal to 220 H/m^2.

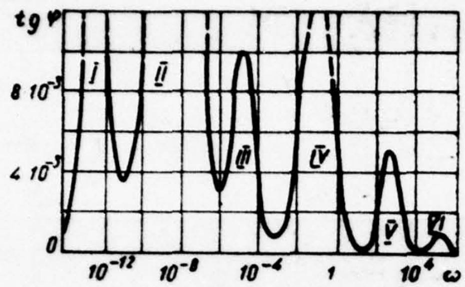

Fig. 7. The relaxation spectrum of metals at 293° K. The dissipation of energy is caused: I - by presence of the pairs of atoms with different atomic radii; II - by viscous flow on grain boundaries; III - by viscous flow in the "amorphous" regions, introduced by plastic deformation (slip band); IV - by diffusion of interstitial atoms; V - by transverse thermal conductivity with the bend of specimen/sample; VI - by intercrystalline thermal conductivity.

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From a quantity of pores in material, depend its elasticity characteristics. The majorities of high-melting materials obtain at present by the methods of powder metallurgy. By these methods it is difficult to manufacture articles made of refractory metals, but that it is more of the refractory compounds, in compact (nonporous) state. Usually refractory metals (tungsten, etc.) have residual porosity not lower than 2-3o/o, and refractory compounds - are not lower than 5-7o/o.

In industry is utilized a series of articles (oilless bearings, filters, etc.), which possess special properties only because of porosity. The data on high-melting materials, the given without indication porosities, cannot have practical application/use. For many refractory compounds there is no information about elasticity characteristics of material in compact state. Values E, G and μ of these materials with zero porosity can be obtained only via extrapolation.

The effect of porosity on elasticity characteristics of materials was studied theoretically and experimentally by many researchers. Theoretically this question studied Mackenzie [84], Gatto [75], M. Yu. Bal'shin [4] and V. V. Skorokhod [55] (for a polyphase mixture).

Mackenzie proposed for Young's modulus of poriferous bodies this expression:

$$\frac{E}{E_0} = 1 - 15P\frac{1-\mu}{7-5\mu} + AP^2, \qquad (24)$$

where P - a porosity; A - constant, determined experimentally; E and E_0 - modulus of elasticity of respectively poriferous and compact body. In expression (24), it is assumed that the Poisson ratio does not depend on porosity.

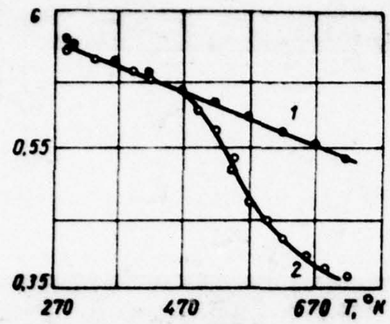

Fig. 8. The curves of the dependences of shear modulus for single-crystal (1) and polycrystalline (2) aluminum on the temperature (along the axis of ordinates is plotted the square of frequency, which is proportional to shear modulus).

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V. V. Skorokhod proposed more general formulas for determining the moduli of elasticity of polyphase systems:

$$\sum_{(i)} \frac{3K+4G}{3K_i+4G}\cdot\frac{K_i}{K}P_i = 1; \qquad (25)$$

$$\sum_{(i)} \frac{5(3K+4G)\,G_iP_i}{(9K+8G)\,G+6G_i(K+2G)} = 1, \qquad (26)$$

where K - a bulk modulus; index i is related to the properties of phases.

Young's modulus can be determined according to the known expression

$$E = \frac{9KG}{3K + G}. \quad (27)$$

Experimental investigations on oxide of aluminum carried out by Coble and Kingery [74]. The obtained by them experimental dependences of elasticity characteristics on porosity satisfactorily coincide with Mackenzie's theoretical dependences. The satisfactory agreement of experimental data with the results of calculation according to expression (24) obtained by Sh. N. Plyatt, Yu. M. Rappoport and Ye. G. Chofnus [49] also on oxide of aluminum. For calculating of Young's modulus abrasive-ceramic materials, they proposed this expression, after assuming $\mu \approx 0.3$:

$$\frac{E}{E_0} = 1 - 1{,}91\, P + AP^2. \quad (28)$$

Value AP^2 can be disregarded, if $P < 0.5$.

Figures 9 shows a change of Young's modulus in dependence on porosity P in specimen/samples made of restored/reduced and electrolytic iron. Extrapolation of the values of Young's modulus of this iron for zero porosity will give value of E, equal approximately to Young's modulus of cast iron. Data will agree well with Mac-Adam's empirical expression

$$\frac{E}{E_0} = (1 - P)^m, \quad (29)$$

where a - an empirical coefficient.

A. B. Lyashchenko, P. I. Melnichuk and I. N. Frantsevich [35] investigated the influence of porosity on Young's modulus of different refractory compounds (Fig. 10). To account for the effect of porosity on Young's modulus for refractory compounds TiB_2, $TiSi_2$, $MoSi_2$, Mo_3Si, and also $ZrSi_2$, VC, TiC, W_2C you suggested the expression

$$E = E_0\left[1 - a\left(\frac{P}{100}\right)^{a_1}\right], \qquad (30)$$

while to Young's modulus of nonporous body - expression

$$E_0 = E_0'\left[1 + a\left(\frac{P}{100}\right)^{a_1}\right], \qquad (31)$$

where

$$E_0' = \frac{E}{1 - \left(\frac{P}{100}\right)^{\frac{2}{3}}}; \qquad (32)$$

a and a_1 - empirical coefficients whose values are different in different compounds.

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In the majority of works, indicated above was studied the effect of porosity on Young's modulus, and about the effect of porosity on modulus of shear and Poisson ratio, were made only different

assumptions. I investigated the influence of porosity on modulus of shear and Poisson ratio [3]. Experiments were conducted in specimen/samples made of poriferous iron. Specimen/samples made of the iron powder of the mark/brand of APZhMA with a diameter of 8 mm and length of 100 mm were sintered at temperature of 1420° K for three hours in the medium of hydrogen.

Young's modulus and displacement were determined simultaneously in each specimen/sample in the resonance frequencies of the transverse and torsional oscillations (in detail this procedure was described in Chapter II). Poisson ratio was calculated according to dependence (4). The obtained experimental data on the infauence of porosity on elasticity characteristics of iron are shown on Fig. 9, where each point is obtained via the averaging of the results of measurement E and G in five specimen/samples.

With an increase in the porosity of Poisson's specimen/samples, it is decreased, moreover the character of its change in the same basic as in the module/moduli of the first and second kind. Thus, assumptions about the equality of Poisson ratio of poriferous and compact material are faulty ones. Extrapolation of the obtained values of Poisson ratio for zero porosity gives $\mu \approx 0.27$, which corresponds to literature data.

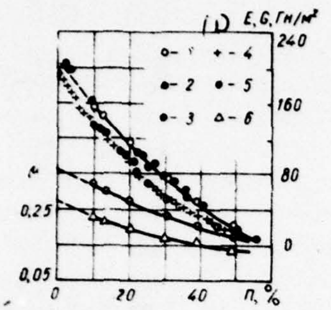

Fig. 9.

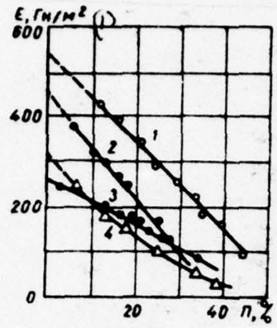

Fig. 10.

Fig. 9. Influence of porosity on elasticity characteristics of iron. Young's modulus E: 1 - cur data: 2, 3 - O. A. Chekhova's data [69]; 4 - McAdam's data [83]. Mcdulus of shear G (5) and Poisson ratio μ (6) - our data.

Key: (1). H/m².

Fig. 10. Influence of porosity cn Young's mcdulus of refractory compounds: 1 - TiB_2; 2 - $MoSi_2$; 3 - $TiSi_2$; 4 - Mo_3Si.

Key: (1). H/m².

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Chapter II.

METHODS OF DETERMINING THE MODULI OF ELASTICITY OF MATERIALS AT HIGH TEMPERATURES.

1. Static method.

With the static method of determining the moduli of elasticity the specimen/sample of material is subjected the effect of static load, is measured its deformations, and then is designed Young's modulus from the relationship/ratio between them. At high temperatures for measuring Young's modulus of material by this method are utilized most frequently the specimen/samples in the form of bars. Specimen/sample is subjected bend in such a way that the load affects on its middle part at one or two points [26] (Fig. 11). Sagging/deflection is measured halfway specimen/sample. Young's modulus can be calculated according to these expressions:

if load is applied at one point,

$$E=\frac{Pl^3}{48Iy};\qquad(33)$$

if load is applied at two points,

$$E=\frac{Pa}{24Iy}(4a^2-3l^2),\qquad(34)$$

where I - moment of inertia; y - sagging/deflection halfway of specimen/sample.

Expressions (33) and (34) are valid only for specimen/samples in which the ratio of length to thickness equal to 10. For shorter specimen/samples it is necessary to introduce the appropriate corrections.

The load application on specimen/sample can be carried out pressing or pulling rod. Load on specimen/sample affects through prisms from refractory materials: tungsten, carbide of silicon, oxide of aluminum. The sagging/deflections of specimen/sample are measured by indicator by the means of rods from sapphire or vitreosil.

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Sometimes sagging/deflection is measured not only halfway, but also at the ends of the specimen/sample in order to consider the thermal

expansion and other factors, calling zero drift. For obtaining the uniform results loading they conduct with the speed within limits of 35-140 N/mm²•min. The installation diagram for measuring Young's modulus [26] is given tc Fig. 12.

The determination cf shear modulus by static method at high temperatures is produced in cylindrical specimen/samples. Specimen/sample is subjected torsion and is measured angle of twist.

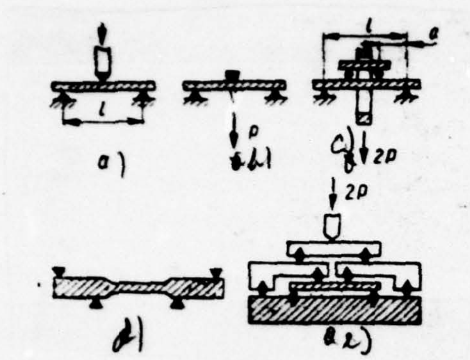

Fig. 11. The schematics of the loading of specimen/sample during the determination of Young's modulus by the static method: a, b) - load is applied at one point; c, d, e) - load is applied at two points.

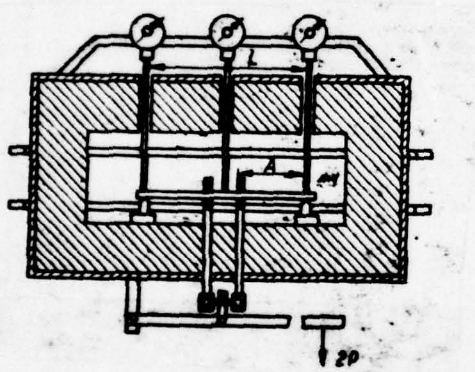

Fig. 12. Installation for determining Young's modulus by static method at high temperatures.

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Shear modulus can be calculated on the formula

$$G=\frac{32Ml}{\pi d^4\theta}, \qquad (35)$$

where M - the torsional moment; l, d - length and diameter of the working part of the specimen/sample; θ - angle of twist.

In installations for determining the modulus of shear [26] (Fig. 13) are applied the massive grip/captures and other devices, in order to avoid emergence in the specimen/sample of bending stresses.

Since the relaxation time is decreased with an increase in the temperature, the modulus of elasticity, determined by static method, will be relaxed. The degree of the relaxation of the modulus of elasticity depends to a considerable extent on specific test conditions: the speed of loading, accuracy/precision of the maintaining of load and temperature. All this leads to the large scatter of the values of the modulus of elasticity. Therefore static method is utilized at present rarely, and measurements conduct by dynamic method.

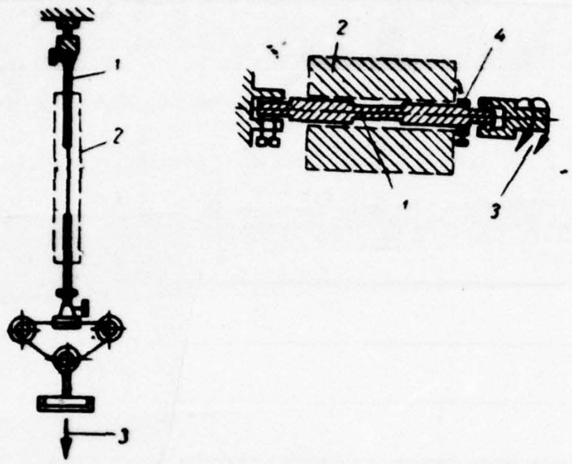

Fig. 13. The installaticn diagrams for determining the shear modulus by static method at higt temperatures [26]: 1 - specimen/sample; 2 - furnace; 3 - load; 4 - indicator, forced against specimen/sample.

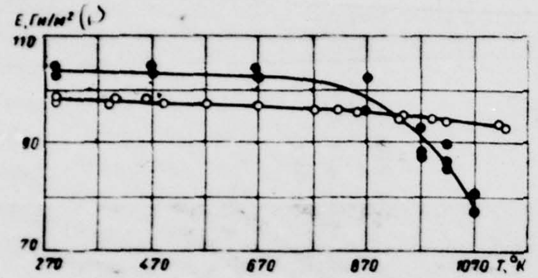

Fig. 14. Temperature dependences of Young's modulus of Pyroceram, obtained by static (dark points) and dynamic (bright points) methods of measurements.

Key: (1). H/m².

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Figure 14 shows the temperature dependences of the modulus of elasticity of glass and Pyroceram, obtained by static [51] and dynamic (our data) methods. To temperature of 670° K, the character of dependence is identical. The small disagreements of absolute values are caused, obviously, by the fact that were undertaken glasses of different boilings. However, at temperature it is higher than 670° K value of Young's modulus, obtained by static method, sharply they are decreased, and the scatter of points increases.

2. Dynamic method.

With the dynamic method of determining the moduli of elasticity, cyclic loads are applied to specimen/sample at a high speed, so that relaxation processes can occur only at very high temperatures. The measurement of the moduli of elasticity is based on what the velocities of propagation in solid of different wave modes are different and depend on elasticity characteristics.

For determining the moduli of elasticity, are measured the values, connected with the velocity of propagation of elastic disturbance/perturbation, transit time and frequency, and also angles, at which is spread this disturbance/perturbation, or is

determined the degree of its effect on the course of other phenomena. Figure 15 gives the frequency bands included by different methods of measuring the module/moduli of elasticity [29]. In pulse installations (Fig. 16) most frequently is utilized the principle of the measurement of the time of propagation of elastic disturbance/perturbation. In test specimen are sent through the specific time interval the momentum/impulse/pulses of high-frequency longitudinal or transverse vibrations. Directly is measured the transit time of this momentum/impulse/pulse (the "train" of waves) through the specimen/sample. To the end/faces of specimen/sample 6, they adhere the quartz plates of this section/shear that during the supplying on them of the voltage in specimen/sample would appear longitudinal or shear waves.

Fig. 15. Frequency bands included by different methods of the measurements of the moduli of elasticity. I - static; II - dynamic; a). the torsion pendulum; b) - the oscillation of a rod and plates; c) - compound/composite resonator-vibrators; d) - ultrasonic waves.

Key: (1). frequency ϕ, per/s.

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Plate 7 is vibration exciter, while plate 5 - receiver. The output/yield of HF generator 1 is controlled by the generator of the pulse repetition frequency 2, which converts continuous wave into the periodically repeated trains. Electrical oscillation/vibrations are converted by the quartz plate into mechanical ones. After achieving opposite end, wave train partially transfer/converts into receiving plate, and it is partially reflected conversely. In receiver mechanical oscillation/vibrations are converted into electrical ones and, in passing by rectifying amplifier 4, they enter cathode-ray oscilloscope 3. At shield they appear in the form of the direct/straight vertical line whose value is proportional to the

amplitude of mechanical wave train in specimen/sample. The train reflected goes conversely, is reflected from end/face and again goes to opposite end/face, etc. In this case, on the shield of oscilloscope, are observed the equidistant lines, which decrease along the length in proportion to the attenuation of waves, since horizontal timing axis shows time, and scan/development itself is synchronized by the pulse repetition frequency. Time t of the passage tandem of the waves of the length l of specimen/sample twice corresponds to scale to the distance between two adjacent lines. Wave propagation velocity

$$c = \frac{2l}{t}. \quad (36)$$

Knowing the velocity of propagation of longitudinal elastic wave in stem, is determined Young's modulus:

$$c_{np} = \sqrt{\frac{E}{\rho}}, \quad (37)$$

where ρ - material density of rod.

On the velocity of propagation of torsion waves, it is possible to determine shear modulus:

$$c_{np} = \sqrt{\frac{G}{\rho}}. \quad (38)$$

Since the piezoelectric converters cannot work at the high temperatures, pulse of installation in essence are utilized for

determining the moduli of elasticity at normal and low temperatures. However, in recent years were developed the installations, making it possible to determine the moduli of elasticity by dynamic method, also, at high temperatures.

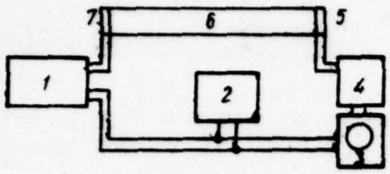

Fig. 16. Diagram of pulse installation for determining the module/moduli of elasticity [27].

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So, B. A. Kalugin and N. G. Mikhaylov [19] created the installation during which it is possible to determine the modulus of elasticity by pulse method to 3000° K. The specimen/sample being investigated with a diameter of 12 mm and with a length of 50 mm has thickenings at the ends by which it is fastened in the dismountable/release water-cooled clamps, that use simultaneously by current supplies. Specimen/sample is heated by the direct transmission of electric current. The intense cooling of the ends of the specimen/sample makes it possible to utilize the piezo converters, attached to end/faces. By B. A. Kalugin and N. G. Mikhaylov is developed the procedure of the computation of the moduli of elasticity on measurements in unevenly heated specimen/sample [19].

At present for determining of elasticity characteristics at high temperatures, most widely are utilized resonance device. Their

operating principle is instituted on the determination of the natural frequency of the oscillating test sample from resonance onset during its agreement with the frequency of vibration exciter. Resonance frequency $f_{рез}$ is connected with the natural frequency of specimen/sample by the dependence

$$f_{рез} = f_{соб}\left(1 - \frac{\delta^2}{8\pi^2}\right), \qquad (39)$$

where δ - a logarithmic decrement of oscillation/vibrations. During the low dissipation of energy in materials, for example with $\delta = 10^{-3}$, it is possible to count $f_{рез} \approx f_{соб}$ with an accuracy to 0.01o/o.

On the relationship/ratio between the natural frequency of specimen/sample, its geometric dimensions and weight, are calculated the elasticity characteristics. For determining the elastic modulus E, are utilized longitudinal or transverse vibrations, while for determining the modulus of shear G, - torsion.

Are given below the basic laws, which exist during the longitudinal, torsion and flexural vibrations of rods. It is assumed that the specimen/samples have a form of fine/thin rectilinear rods of uniform section/cut, moreover in them λ >> d, where λ - wavelength of the exited in specimen/sample vibration, and d - a transverse size/dimension of specimen/sample.

Longitudinal oscillations. With longitudinal oscillations the axle/axis of rod remains fixed, and cross sections oscillate in the direction, perpendicular to their planes. It is assumed that during the longitudinal oscillations of prismatic rod its cross sections remain flat/plane, and the particles, which lie at these section/cuts, accomplish motions only in the direction of the axle/axis of rod. Longitudinal tensile strains and compression with oscillations of rod are accompanied by some lateral deformations, however, when the length of longitudinal waves is great in comparison with the transverse size/dimensions of rod, lateral deformations they usually disregard.

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If necessary it is possible to introduce correction according to the method, proposed by Rayleigh.

For the longitudinally oscillating rod (Fig. 17) let us introduce the following designations: u - the longitudinal travel of the arbitrary cross section of rod, which is located at a distance of x from the origin of coordinates. This displacement/movement is the function of coordinate x and time t; ε - elongation per unit length; S - cross-sectional area; F - longitudinal tensile force, $F=SE\varepsilon$; ϱ — material density of rod; l - length of rod.

Elongation per unit length and tensile force in arbitrary cross section at a distance of x from the origin of coordinates accepted at the end of the rod can be represented in the form

$$\varepsilon = \frac{\partial u}{\partial x}; \; F = SE\frac{\partial u}{\partial x}.$$

For section/cut at a distance dx from the first tensile force $F + dF = SE\left(\frac{\partial u}{\partial x} + \frac{\partial^2 u}{\partial x^2}dx\right)$. The force of inertia of element of length with a of dx, the locating between the section/cuts indicated rod, will be $-S\rho\, dx\frac{\partial^2 u}{\partial t^2}$.

Applying d'Alembert's principle to the elementary part of the oscillating rod in question with a length of dx, we obtain the differential equation of longitudinal oscillations of a rod in the form $-S\rho\frac{\partial^2 u}{\partial t^2} + SE\frac{\partial^2 u}{\partial x^2} = 0$ or

$$\frac{\partial^2 u}{\partial t^2} = a^2\frac{\partial^2 u}{\partial x^2}, \qquad (40)$$

where

$$a^2 = \frac{E}{\rho}. \qquad (41)$$

Since displacement/movement u depends on coordinate x and time t, it can be presented as u = XZ, where X - certain function only of coordinate x; Z - certain function only of time t.

Functions X, Z must be such that would be satisfied equation (40).

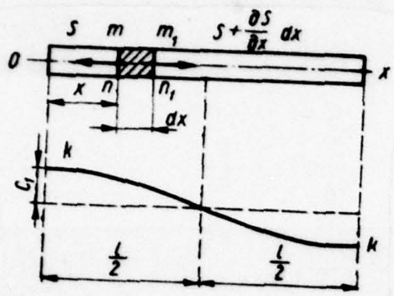

Fig. 17. Rod, which acccmplishes lcngitudinal cscillations.

Eage 23.

If rod accomplishes oscillations of one of its cwn forms whose frequency is equal to $\omega/2\pi$, then the soluticn of equation (40) takes the form

$$u=X(A\cos\omega t+B\sin\omega t). \qquad (42)$$

where A and B - integraticn constant; ω - angular frequency; function X determines the form of oscillaticns and is called normal function.

With oscillations cf rod with free ends, the tensile force at ends must be equal to zero, which answers the fcllowing conditions at the ends:

$$\left(\frac{\partial u}{\partial x}\right)_{x=0}=0;\ \left(\frac{\partial u}{\partial x}\right)_{x=l}=0. \qquad (43)$$

Substituting expression (42) in equation (40), we obtain $-\omega^2X=$

$a^2(d^2X/dx^2)$. In that case

$$X = C\cos\frac{\omega x}{a} + D\sin\frac{\omega x}{a}. \qquad (44)$$

In order to satisfy first condition (43), it is necessary to place D = 0, second condition (43) will be satisfied, if

$$\sin\frac{\omega l}{a} = 0. \qquad (45)$$

Equation (45) is the frequency equation for the case in question, which makes it possible to calculate the frequencies of its own forms of longitudinal oscillations of a rod with free ends. Frequency equation will be satisfied with

$$\frac{\omega l}{a} = i\pi, \qquad (46)$$

where i - integer.

Setting i = 1, 2, 3, ..., we obtain the frequencies of the various forms of oscillations. The angular frequency of the basic form of oscillations will be obtained with substitution i = 1. Then

$$\omega_1 = \frac{a\pi}{l} = \frac{\pi}{l}\sqrt{\frac{E}{\rho}}. \qquad (47)$$

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The corresponding period of oscillations $T_1 = 2\pi/\omega_1 = 2l\sqrt{\rho/E}$ or, bearing in mind that $\sqrt{E/\rho}$ - velocity of propagation longitudinal

elastic wave in stem,

$$T_1 = \frac{2l}{c}. \quad (48$$

Frequency of the first form of the natural oscillations

$$f_{np} = \frac{1}{2l}\sqrt{\frac{E}{\rho}} \quad (49)$$

or

$$f_{np} = \frac{C}{2l}. \quad (50)$$

The form of this form of oscillations is represented in Fig. to 17 curves kk whose ordinates are determined from the equation

$$X_1 = C_1 \cos\frac{\omega_1 x}{a} = C_1 \cos\frac{\pi x}{l}. \quad (51)$$

From the given above elementary unpacking/facings escape/ensue several derivations, which have important value for experimental determination of elastic modulus E.

1. Elastic modulus E can be determined by resonance frequency of first form of longitudinal oscillations of test sample made of expression (50)

$$E = 4l^2 \rho f_{np}^2. \quad (52)$$

2. During oscillations of a rod on first (basic) form of oscillations from formula (51) it follows that $X_1 = 0$ with $x = l/2$, i.e., average

along the length of rod section/cut is motionlessly vibration node, which makes it possible during construction of installation to utilize mean section of specimen/sample for its attachment during tests, after leaving ends free.

3. Frequencies of high forms of longitudinal oscillations of test sample are more frequency of first form into 2, 3, 4, 5, ..., times, which makes it possible during measurements to differ resonance frequencies of longitudinal oscillations of specimen/sample from different interference/jammings.

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Sometimes the test sample, which varies on the basic form of oscillations, is called half-wave, since wavelength $\lambda = \frac{c}{f_{np}}$, while since for fundamental frequency $f_{np} = \frac{c}{2l}$, that after substitution f_{np} into the formula indicated we obtain $\lambda = 2l$ or $l = \lambda/2$.

Torsional oscillations. During torsional oscillations the axle/axis of rod remains fixed, and its cross sections are rotated around it. It is assumed that during torsional oscillations the cross sections remain flat/plane, and radii of these cross sections - by straight lines. For the rod, which accomplishes torsional oscillations (Fig. 18), θ - the angle of rotation of arbitrary cross

section, which is located at a distance of x from the origin of coordinates, is the function of time t; G - modulus of shear of the material of rod; I_p - polar moment of the inertia of the cross section of rod; M - torsional moment in section/cut on distance x from the origin of coordinates $\left(M = GI_p \frac{\partial\theta}{\partial x}\right)$; ρ — material density of rod.

The torsional moment in section/cut at a distance x+dx from the origin of coordinates will be

$$M + \frac{\partial M}{\partial x} dx = GI_p \frac{\partial\theta}{\partial x} + \frac{\partial}{\partial x}\left(GI_p \frac{\partial\theta}{\partial x}\right) dx.$$

The moment of the forces of inertia of the cell/element of rod with a length of dx is equal to $\rho I_p \frac{\partial^2\theta}{\partial t^2}$.

Applying d'Alembert's principle, we obtain the differential equation of motion of cell/element dx:

$$\rho I_p \frac{\partial^2\theta}{\partial t^2} = GI_p \frac{\partial^2\theta}{\partial x^2}. \tag{53}$$

or, designating $G/\rho = a^2$, we obtain

$$\frac{\partial^2\theta}{\partial t^2} = a^2 \frac{\partial^2\theta}{\partial x^2}. \tag{54}$$

Equation (54) by nature completely coincides with equation (40); therefore the obtained above dependences for the longitudinally oscillating rod can be used for the rod, which accomplishes torsional oscillations.

Fig. 18. Rod, which accomplishes torsional oscillations.

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From that given it is possible to make such conclusions.

1. Modulus of shear G can be determined by resonance frequency of first form of torsional oscillations:

$$G = 4\rho l^2 f^2_{\text{кр}}. \qquad (55)$$

2. During torsion oscillations of a rod fixed is its mean section. Therefore in the case of fastening rod during the wire suspensions, arrange/located on its end/faces, it is necessary to utilize wire of minimum thickness. Otherwise the mass of wire can considerably affect resonance frequency.

3. Frequencies of high forms of torsional oscillations of test sample are more frequency of first form into 2, 3, 4, 5, ..., times, which makes it possible during measurements to differ resonance frequencies of torsional oscillations from resonance frequencies of

transverse vibrations and interference/jammings.

Transverse vibrations. With this mode, the axle/axis of rod experience/tests bend in one plane, and its cross sections participate in forward motion in the plane of bending and in rotary motion around the axle/axes, perpendicular to the plane of bending.

Figures 19 shows the transverse forces and the bending moments, which effect on the cell/element of rod dx during transverse vibrations. Besides transverse forces on the cell/element of rod dx, affects another inertial force, equal to $\rho S \frac{\partial^2 y}{\partial t^2} dx.$

The equation of transverse oscillations of a rod, which gives satisfactory accuracy/precision for long stems, can be obtained, if is considered only forward motion of cell/element dx. In this case are totaled the transverse forces and the inertial forces, which effect on cell/element dx, which gives

$$\frac{\partial Q}{\partial x} = \rho S \frac{\partial^2 y}{\partial t^2}. \qquad (56)$$

If the cross sections of rod are low in comparison with its length, then the differential equation of elastic line takes the form

$$EI \frac{\partial^2 y}{\partial x^2} = -M, \qquad (57)$$

where I - moment of the inertia of the cross section of rod relative to neutral axle/axis, section/cut, perpendicular to vibration plane; M - bending moment in any cross section.

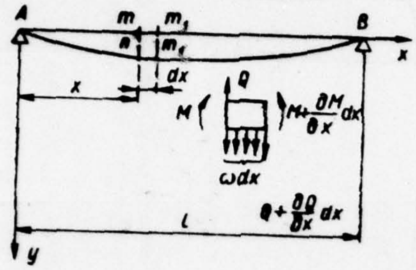

Fig. 19. Rod, which accomplishes transverse vibrations.

Page 27.

It differentiated this expression twice, we will obtain

$$\frac{\partial^2}{\partial x^2}\left(EI\frac{\partial^2 y}{\partial x^2}\right) = -\frac{\partial Q}{\partial x}. \qquad (58)$$

After substituting value of $\frac{\partial Q}{\partial x}$ from equation (56), we will obtain the common/general/total equation of transverse oscillations of a rod in the form

$$EI\frac{\partial^4 y}{\partial x^4} = -\rho S\frac{\partial^2 y}{\partial t^2}. \qquad (59)$$

or, after introducing the designations

$$a^2 = \frac{EI}{S\rho}, \qquad (60)$$

in the form

$$\frac{\partial^2 y}{\partial t^2} + a^2\frac{\partial^4 y}{\partial x^4} = 0. \qquad (61)$$

If rod accomplishes harmonic oscillation, then sagging/deflection in any place can be represented as the equation

$$y = X(A\cos\omega t + B\sin\omega t), \tag{62}$$

where X - a function of coordinate x, which determines the normal form of oscillations.

Substituting expression (62) in equation (61), we obtain

$$\frac{\partial^4 X}{dx^4} = \frac{\omega^2}{a^2}X. \tag{63}$$

If we designate

$$\frac{\omega^2}{a^2} = \frac{\omega^2 S\rho}{EI} = k^4, \tag{64}$$

then sin kx, cos kx, sh kx, ch kx they will be the particular solutions of equation (63), and its general solution it will be

$$X = C_1(\cos kx + \operatorname{ch} kx) + C_2(\cos kx - \operatorname{ch} kx) + C_3(\sin kx + \operatorname{sh} kx) + C_4(\sin kx - \operatorname{sh} kx). \tag{65}$$

where C_1, C_2, C_3, C_4 - the constants which are determined in each specific case from conditions at the ends of the rod.

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The specimen/sample, suspend/hung from suspensions in vibration nodes, is rod with free ends. In this case the bending moment and

transverse force are equal to zero at free end, i.e.,

$$\left(\frac{d^2 X}{\partial x^2}\right)_{x=0} = 0;\quad \left(\frac{d^3 X}{dx^3}\right)_{x=0} = 0; \qquad (66)$$

$$\left(\frac{\partial^2 X}{\partial x^2}\right)_{x=l} = 0;\quad \left(\frac{\partial^2 X}{\partial x^3}\right)_{x=l} = 0. \qquad (67)$$

From condition (66) it follows that $C_2 = C_4 = 0$, then the general solution will take the form

$$X = C_1(\cos kx + \operatorname{ch} kx) + C_3(\sin kx + \operatorname{sh} kx). \qquad (68)$$

According to conditions (67) let us have:

$$C_1(-\cos kl + \operatorname{ch} kl) + C_3(-\sin kl + \operatorname{sh} kl) = 0; \qquad (69)$$
$$C_1(\sin kl + \operatorname{ch} kl) + C_3(-\cos kl + \operatorname{ch} kl) = 0.$$

Different from zero solutions for the constants C_1 and C_3 will be obtained only in such a case, when the determinant of system of equations (69) is equal to zero:

$$(-\cos kl + \operatorname{ch} kl)^2 - (\operatorname{sh}^2 kl - \sin^2 kl) = 0, \qquad (70)$$

while since $\operatorname{ch}^2 kl - \operatorname{sh}^2 kl = 1$; $\cos^2 kl + \sin^2 kl = 1$, then frequency equation takes the final form

$$\cos kl \operatorname{ch} kl = f. \qquad (71)$$

First six roots of this equation such: $k_1 l = 0$; $k_2 l = 4{,}730$; $k_3 l = 7{,}853$; $k_4 l = 10{,}996$; $k_5 l = 14{,}137$; $k_6 l = 17{,}279$.

Frequencies corresponding to these roots, are calculated from

expression (65):

$$f_1 = 0;\ f_2 = \frac{\omega_2}{2\pi} = \frac{k_2^2 a}{2\pi};\ f_3 = \frac{\omega_3}{2\pi} = \frac{k_3^2 a}{2\pi};\ldots \qquad (72)$$

Substituting the roots of equation (71) in equation (70), is obtained ratio C_1/C_3 for the appropriate modes, and from equation (68) is found the form of elastic line during oscillations. The first form of oscillations, which corresponds to the first different from zero frequencies, has two assemblies at a distance of 0.224 l from the ends of the rod. Figure 20 shows the forms of the elastic line of rod during transverse vibrations and the position of nodes.

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On the basis of that presented, it is possible to draw the conclusions:

1. Young's modulus can be determined by the resonance frequencies of the various forms of transverse oscillations of a rod according to formulas (72) and (61). For the first form

$$E = \frac{4\rho S}{I}\left(\frac{\pi l^2}{4{,}730^2} f\right)^2. \qquad (73)$$

2. Filaments of suspensions must be furnished near nodes which with first form are located at a distance by 0.224 l from ends of specimen/sample.

3. Frequencies of high forms of transverse vibrations, as follows of expressions (72), more frequency of first form into 2.76; 5.41; 8.94; ... once, which makes it possible to differ resonance frequencies of transverse vibrations from resonance frequencies of torsional oscillations and interference/jammings.

Determining the moduli of elasticity at high temperatures, it is necessary to keep in mind that the linear dimensions and the specific gravity/weight of specimen/sample change during the heating:

$$l_t = l(1+\alpha t); \tag{74}$$

$$d_t = d(1+\alpha t); \tag{75}$$

$$\rho_t = \frac{\rho}{(1+\alpha t)^3}, \tag{76}$$

where l, d and ρ - respectively length, diameter and density of specimen/sample at room temperature; l_t, d_t and ρ_t - respectively length, diameter and density of specimen/sample at temperature t; α - coefficient of the linear expansion of the material of specimen/sample.

Taking into account formula (74), (75) and (76), it is possible to calculate the moduli of elasticity at high ones the temperature:

during the longitudinal oscillations

$$E_t = 4\rho l^2 f_{np}^2 (1 + \alpha t)^{-1}; \tag{77}$$

during transverse vibraticns

$$E_t = \frac{4\rho S}{I}\left(\frac{\pi l^2}{4{,}730^2} f_{\text{кр}}\right)^2 (1+\alpha t)^{-1}; \qquad (78)$$

during torsicnal oscillaticns

$$G = 4\rho l^2 f_{\text{кр}} (1+\alpha l)^{-1}. \qquad (79)$$

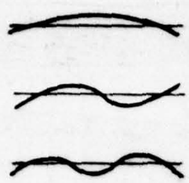

Fig. 20. Forms of the elastic line of rod during transverse vibrations.

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3. Determination of the moduli of elasticity during the uneven heating of specimen/sample.

Use of uneven heating during the measurements of the moduli of elasticity at high temperatures gives a series of advantages. In this case the testing unit can be simpler, are improved the conditions of the work of exciter and receiver of the oscillations, arrange/located on the end/faces of specimen/sample less heated, than its middle.

The dynamic method of determining elasticity characteristics from resonance frequency of specimen/sample makes it possible to use the uneven heating during which the middle of specimen/sample is heated more strongly than its ends, as a result of the special feature/peculiarities of the oscillating processes which occur in

specimen/sample.

The procedure of the determination of Young's modulus from the resonance frequency of the longitudinal oscillations of specimen/sample during his uneven heating is developed by V. A. Kuz'menko [30]. The essence of this procedure consists in following.

In the unevenly heated along the length specimen/sample of the value of Young's modulus, they are different on different sections. By experiments and subsequent calculations it was establish/installed, that a change in Young's modulus along the length of specimen/sample when the middle of specimen/sample was heated more than its end/faces, is expressed well by the parabolic dependence

$$E(x)=E_0+\eta x^2. \quad (80)$$

where E_0 - value of Young's modulus in the vibration node which is located the halfway unevenly heated specimen/sample.

The origin of coordinates places in the vibration node of specimen/sample. For determining coefficient η is utilized the fact that the curves of temperature dependence E during the uniform and uneven heating of specimen/sample, differ little one from another (Fig. 21). These data were obtained during installation [31],

permitting implemention of both uniform and uneven heating of specimen/sample. During uniform heating the temperature of the end/faces of specimen/sample differed from the temperature of its central part not more than to 3o/o.

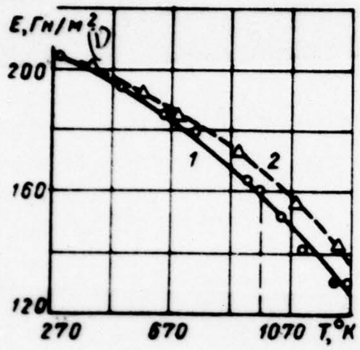

Fig. 21. Dependence of Young's modulus of the alloy EI612 on temperature, obtained during the uniform tc (1) and uneven (2) heating of specimen/sample along the length (longitudinal oscillation).

Key: (1). H/m².

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During the uneven heating of specimen/sample, the temperature of specimen/sample differed to 50o/o from the temperature of its junction/unit section/cut.

Knowing the half of the length of specimen/sample and the temperature of its middle and end/faces, through dotted curve find approximate values E_0 and E_1, but through them, utilizing expression (81), coefficient η.

During the derivation of the differential equation of longitudinal oscillations of a rod, which has variable along the length Young's modulus, one should consider that the expressions for a tensile force at a distance of x from the beginning of coordinates and force of inertia of the cell/element of rod with a length of dx will take the same form as rod with constant module/modulus.

Tensile force in section/cut at a distance x+dx from the origin of coordinates will be

$$F+dF=SE\frac{\partial u}{\partial x}+\left(S\frac{\partial E}{\partial x}\cdot\frac{\partial u}{\partial x}+SE\frac{\partial^2 u}{\partial x^2}\right)dx.$$

Then differential equation of longitudinal oscillations of a rod, which has alternating/variable along the length module/modulus, can be recorded in the form

$$E\frac{\partial^2 u}{\partial x^2}+\frac{\partial E}{\partial x}\cdot\frac{\partial u}{\partial x}-\frac{\rho\partial^2 u}{\partial t^2}=0. \quad (81)$$

Substituting in equation (81) value of u from expression (39), we obtain

$$E\frac{\partial^2 X}{\partial x^2}+\frac{\partial E}{\partial x}\cdot\frac{\partial X}{\partial x}+\omega^2\rho X=0. \quad (82)$$

The solution of equation (82) will be the formula

$$\psi(x)=\gamma(x-Ax^3+Bx^5-Cx^7+\ldots), \quad (83)$$

where γ - constant, and A, B, C, ... - the coefficients in which they enter E_0, η, ρ and frequency f, measured at the which interests us temperature of the junction/unit section/cut of specimen/sample.

In this case will occur the boundary condition

$$\psi'\left(\frac{1}{2}\right)=0. \quad (84)$$

Determining ψ' from expression (83), we find the equation whose solution will give value E_0. Values E_0, obtained by such calculations, they are shown on Fig. 21 by black/ferrous small circle.

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Since differential equations of torsion and longitudinal oscillations of a rod are similar, determination of shear modulus during the uneven heating of specimen/sample will in no way differ from the determination of Young's modulus under the same conditions.

In the case of transverse vibrations of the unevenly heated rod, the differential equation of oscillations will take the form

$$\frac{\partial}{\partial x^2}\left(E(x)\,I\frac{\partial^2 y}{\partial x^2}\right)+S\rho\frac{\partial^2 y}{\partial t^2}=0, \quad (85)$$

where E(x) - the function, which expresses a change in Young's modulus along the length of specimen/sample.

The procedure of the determination of Young's modulus from the resonance frequency of transverse vibrations during the uneven heating of specimen/sample was developed by V. A. Dreshpak [13]. For obtaining the expression, which makes it possible to determine the natural frequency of rod, it utilized Ritz's method [61].

Applying the method of Ritz, the sagging/deflection of rod during oscillations takes in the form

$$y = X \cos \omega t, \tag{86}$$

where X determines the form of oscillations.

For convenience the origin of coordinates places to the center of gravity of the mean section of rod, and the length of specimen/sample is taken as equal to $2l$. Greatest potential energy of the rod

$$V = \frac{1}{2}\int_{-l}^{l} EI\left(\frac{d^2X}{dx^2}\right)^2 dx, \tag{87}$$

while the greatest kinetic energy

$$T = \frac{1}{2}\int_0^l S\rho (X\omega)^2 dx. \qquad (88)$$

whence

$$\omega^2 = \frac{I}{S\rho} \cdot \frac{\int_{-l}^{l} E\left(\frac{d^2 X}{\partial x^2}\right) dx}{\int_{-l}^{l} X^2 dx}. \qquad (89)$$

To exact solution for the frequency of the basic form of oscillations corresponds the minimum value of expression (89). For obtaining approximate solution of X, is accepted in the form of the series

$$X = a_1 \varphi_1(x) + a_2 \varphi_2(x) + a_3 \varphi_3(x) + \ldots, \qquad (90)$$

where each of the functions ϕ satisfies conditions at the ends of the rod. Substituting formula (90) in expression (89) and satisfying the condition of the minimum, we obtain

$$\frac{\partial}{\partial a_n} \cdot \frac{\int_{-l}^{l} E\left(\frac{d^2 X}{dx^2}\right)^2 dx}{\int_{-l}^{l} X^2 dx} = 0 \qquad (91)$$

or

$$\int_{-l}^{l} X^2 dx \frac{\partial}{\partial a_n} \int_{-l}^{l} E\left(\frac{d^2 X}{dx^2}\right)^2 dx - \int_{-l}^{l} E\left(\frac{d^2 X}{dx^2}\right)^2 dx \frac{\partial}{\partial a_n} \int_{-l}^{l} X^2 dx = 0. \qquad (92)$$

Utilizing formula (89), we obtain

$$\frac{\partial}{\partial a_n}\int_{-l}^{l}\left[E\left(\frac{d^2X}{\partial x^2}\right)^2-\frac{S\rho\omega^2}{I}X^2\right]dx=0. \qquad (93)$$

Let us now determine such values of the constants a_1, a_2, a_3, ... in expression (90), which turn into the minimum the integral

$$R=\int_{-l}^{l}\left[E\left(\frac{d^2X}{dx^2}\right)-\frac{S\rho\omega^2}{I}X^2\right]dx. \qquad (94)$$

Equations (93) are uniform and linear relative to a_1, a_2, a_3, ... their number is equal to a number of terms into expressions (90). Equalizing to zero determinant of these equations is obtained the frequency equation from which it is possible to calculate the frequencies of the various forms of oscillations.

As function $\Phi(x)$ are accepted the normal functions of prismatic rod with free ends. Such functions give satisfactory approach/approximation for the frequency of the basic form:

$$X_i=C_i(\cos k_ix\,\mathrm{ch}\,k_il+\mathrm{ch}\,k_ix\cos k_il). \qquad (95)$$

For simplification the arbitrary constant is accepted in the form

$$C_i=\frac{1}{\sqrt{\cos^2 k_il+\mathrm{ch}\,k_il}}. \qquad (96)$$

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Normal function which answers first root $k_1 l=0$, it is constant value, equal to $1/\sqrt{2}$, and the corresponding to it motion represents the displacement of rod as solid in the direction of Y axis. Then series (90) can be recorded in the form

$$X=a_1\frac{1}{\sqrt{2}}+a_2\frac{\cos k_2 x \operatorname{ch} k_2 l+\operatorname{ch} k_2 x \cos k_2 l}{\sqrt{\cos^2 k_2 l+\operatorname{ch} k_2 l}}+\cdots. \quad (97)$$

Substituting this expression in equation (93), we obtain

$$\frac{\partial}{\partial a_n}\left\{E_0\int_{-l}^{l}\left(1+a\frac{x^2}{l^2}\right)\sum_{i=1,2,3,\ldots}\sum_{j=1,2,3,\ldots}a_i a_j \varphi_i'' \varphi_j'' dx-\right.$$
$$\left.-\frac{S\rho\omega^2}{I}\int_{-l}^{l}\sum_{i=1,2,3,\ldots}\sum_{j=1,2,3,\ldots}a_i a_j \varphi_i \varphi_j dx\right\}=0. \quad (98)$$

Let us designate

$$\int_{-l}^{l}\left(1+a\frac{x^2}{l^2}\right)\varphi_i'' \varphi_j'' dx=\alpha_{ij};$$

$$\int_{-l}^{l}a_i a_j \varphi_i \varphi_j dx=\beta_{ij};\quad \frac{S\rho\omega^2}{IE_0}=\lambda. \quad (99)$$

Then from equation (98) we find

$$\sum_{i=1,2,3}a_i(\alpha_{in}-\lambda\beta_{in})=0. \quad (100)$$

For determining the basic form of oscillations virtually it is sufficient two members of series (90). In this case equations (100) will take the form

$$a_1(\alpha_{11}-\lambda\beta_{11})+a_2(\alpha_{21}-\lambda\beta_{21})=0;$$
$$a_1(\alpha_{12}-\lambda\beta_{22})+a_2(\alpha_{22}-\lambda\beta_{22})=0. \quad (101)$$

For our case

$$\varphi_1^{\cdot}=0;$$
$$\varphi_2^{\cdot}=k_2^2\frac{-\cos k_2x\,\mathrm{ch}\,k_2l+\mathrm{ch}\,k_2x\cos k_2l}{\sqrt{\cos^2k_2l+\mathrm{ch}^2k_2l}}. \quad (102)$$

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Now we can calculate the values of the coefficients of equation (101)

$$\left.\begin{array}{c}\alpha_{11}=0; \qquad \alpha_{12}=0; \qquad \alpha_{21}=0;\\ \alpha_{22}=\int\limits_{-l}^{l}\left(1-a\frac{x^2}{l^2}\right)(\varphi_2^{\cdot})^2\,dx=\frac{2{,}365^4}{l^3}(1+0{,}3212a);\\ \beta_{11}=l; \qquad \beta_{12}=\beta_{21}=0; \qquad \beta_{22}=l.\end{array}\right| \quad (103)$$

Let us write the determinant of equations (101) and equate it to zero:

$$\begin{vmatrix}-\lambda l & 0\\ 0 & \frac{2{,}365^4}{l^3}(1+0{,}3212a)-\lambda l\end{vmatrix}=0. \quad (104)$$

Hence

$$\lambda=\frac{2{,}365^4}{l^4}(1+0{,}3212\,a) \quad (105)$$

or, if we substitute values λ,

$$\omega=\frac{2{,}365^2\sqrt{1+0{,}3212\,a}}{l^2}\sqrt{\frac{E_0I}{\rho S}}. \quad (106)$$

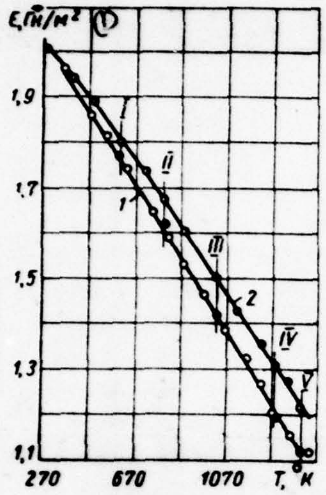

Fig. 22.

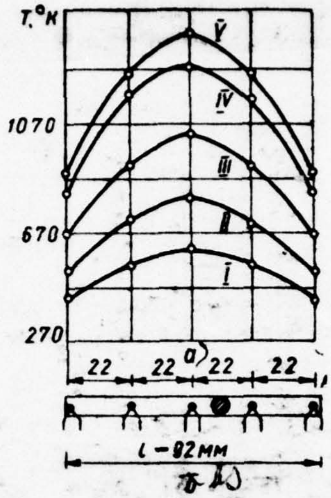

Fig. 23.

Fig. 22. Dependence of Young's modulus steel Kh14N14V2M, obtained during uniform to (1) and uneven (2) heating of specimen/sample along the length (transverse vibration).

Key: (1). H/m².

Fig. 23. Temperature distribution along the length of unevenly heated specimen/sample (a) and diagram of installation of thermocouples (b).

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After substituting into formula (106) $\omega = 2\pi f$ and $L = 2l$, we

will obtain the expression

$$E_0 = \frac{4\rho S}{l}\left(\frac{\pi L^2}{4,73}f\right)^2 \frac{1}{(1+0,3212a)}. \qquad (107)$$

The experimental check of the obtained calculated dependences was carried out by determining Young's modulus of steel Kh14N14V2M in the resonance frequency of transverse vibrations during the uniform and uneven heating of specimen/sample. The temperature of middle and end/faces of specimen/sample during uniform heating differed not more than to 2o/o, but at nonuniform the difference of the temperatures reached 50o/o. Coefficient a determined, as in the case of longitudinal oscillations, according to the curve/graph of the temperature dependence of Young's modulus of the unevenly heated specimen/sample. The calculation of Young's modulus was produced for five values of temperature (I- VI in Fig. 22). The temperature distribution along the length of specimen/sample is shown on Fig. 23 (I - V the same as in Fig. 22). Computed values of Young's modulus (dark points in Fig. 22) coincide well with the values, obtained during uniform heating.

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CHAPTER III.

INSTALLATIONS FOR DETERMINING THE MODULI OF ELASTICITY OF MATERIALS AT NORMAL AND HIGH TEMPERATURES.

1. Determination of the moduli of elasticity at normal temperatures.

For determining of elasticity characteristics of high-melting materials at high temperatures, it is necessary to accurately know their value at normal temperatures. Such measurements are conducted during installations UP-1 and UP-4 [31]. These installations are uncomplicated on device and give the possibility to measure the elasticity characteristics in the test samples of various forms and size/dimensions. The latter fact, and also that in oscillatory systems are absent the additional devices, necessary in high-temperature installations, they make it possible to conduct the measurements of the resonance frequency of specimen/sample more accurately, also, with the small expenditure of time.

During installation UP-1 (Fig. 24) is measured the resonance frequency of the longitudinal oscillations of the test samples with a diameter of 7-8 mm and by length 70-200 mm at room temperature.

As the source of mechanical oscillations serves plate 6 of polarized ceramics of barium titanate, rigidly attached in holder 7. To the electrodes of plate, is supplied the variable-frequency voltage from master oscillator 8, as which is utilized the audiofrequency oscillator of the type ZG-12 or by any other with similar performance data.

Specimen/sample 3, attached in sheet rubber 4, is establish/installed on stand 5, that is moved with the aid of the mechanism of 9 installations of specimen/sample in different directions. The plate of rubber, in which is fastened the specimen/sample, is furnished in the junction/unit section/cut of the latter; the small displacement of plate from the position indicated they do not virtually affect the resonance frequency of specimen/sample. Oscillations from piezoelectric plate 6 are transferred to the specimen/sample through the medium, which fills the interval/gap between the end/face of specimen/sample and the plate.

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If the material of specimen/sample possesses the low damping properties, then even with cutput potential of the master oscillator, which does not exceed 20-50 V, resonance oscillations of specimen/sample easily are excited through the air gap between the end/face of specimen/sample and plate 6. Otherwise it is necessary to raise the stress, applied to electrodes of the plate of exciter, or to reinforce the acoustic contact of specimen/sample with exciter, after filling with liquid the interval/gap between the end/face of specimen/sample and the plate.

The oscillations of specimen/sample are absorbed by the receiving sensor, which are of fine/thin titanate-barium plate 2, fixed to housing installation 1 and the screened foil. The sensor of this construction/design is sufficiently sensitive and accepts the oscillations of the specimen/sample through the air gap between end/face of specimen/sample and foil, stuck on plate 2. If the amplitude of resonance oscillations of specimen/sample is small, this interval/gap is also filled with liquid. In the presence of the air gaps between sensors and end/faces of specimen/sample, the latter can be considered as free rod, and its resonance frequency of

longitudinal oscillations is actually equal to its own; after the filling of interval/gaps with liquid the resonance frequency of specimen/sample is decreased by the tenths of percentage. Signal from receiving sensor is supplied to electron oscillograph EO-7, which is utilized as the marker of resonance. Since the stress, removed from the electrodes plates 2, very weak, in installation is amplifier by 10 for its strengthening before supply to oscillograph. By us is applied amplifier U-2-6, having frequency band 16 Hz - 30 kHz and sensitivity 30 μV.

During a steady change in the frequency of audiofrequency oscillator on oscilloscope face, is accurately visible the torque/moment of resonance onset. Rough reading of frequency is produced on the dial/limb of generator, and precise - with the aid of quartz heterodyne wavemeter with the use of Lissajous figures on the shield of oscilloscope 11 or of electronic frequency meter 12.

Installation UP-4 gives the possibility to determine the module/moduli of the first and second kind according to the resonance frequency of respectively flexural and torsional oscillations. On this installation the excitation of oscillations in specimen/sample and their transmission from specimen/sample are realize/accomplished through the filaments of suspension. The diagrams of the suspensions of specimen/samples are shown on Fig. 25.

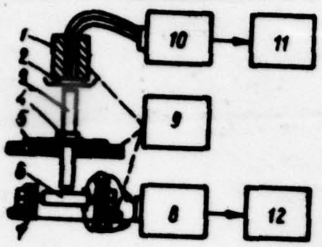

Fig. 24. Block diagram cf installation UP-1.

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According to the diagram, given to Fig. 25a [33, 43], the specimen/sample is hung in the loops of filaments. The longitudinal travel only of one filament cause in specimen/sample flexural vibrations. Through another filament is realize/accomplished recording of resonance frequency. This diagram makes it possible to determine only Young's modulus. The diagram, given to Fig. 25b, was proposed to N. N. Yermolov and E. Kh. Ripp [16]. Specimen/sample has at ends a concentration of mass. Filaments are fastened for the tags, adjustable on the end/faces of specimen/sample. During the longitudinal travel of filament in specimen/sample, are excited flexural and torsional oscillations, which makes it possible to measure Young's modulus and modulus of shear (it is necessary to only consider the effect of the concentrated masses).

According to the diagram, presented in Fig. 25c, flexural and torsional oscillations in specimen/sample are excited through the tangentially welded-on to it filaments. The diagram, similar to this, is applied by K. Syusse [88] for determining the modulus of shear of nickel.

The application/use of diagrams indicated above of suspensions n high-temperature installations is difficult, since welding or soldering of filaments to specimen/samples made of high-melting materials is not always possible, but the majority of them is worked with difficulty, which impedes the installation of tags.

By us are applied the methods of the suspension of specimen/sample in the loops of filaments [24], shown on Fig. 25d and e. With the method of suspension, given to Fig. 25c, the test sample of round cross-section is packed in the loops of filaments. If one branch of suspension accomplishes longitudinal travel, then as a result of friction between the filament and the specimen/sample in it are excited flexural and torsional oscillations. Through another filament by the same way is realize/accomplished recording of the resonance frequencies of the flexural and torsional oscillations. The best effect is obtained when the filament completely encompasses specimen/sample, as shown in Fig. 25e.

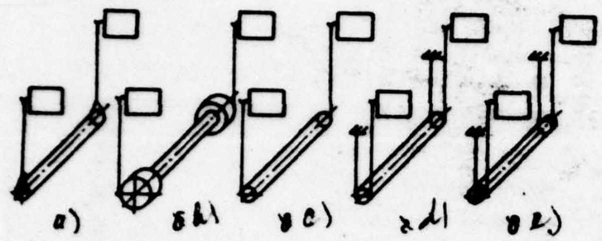

Fig. 25. Diagrams of the suspensions of specimen/samples during the determination of Young's modulus and displacement.

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The used by us method of suspension makes it possible to determine Young's modulus and shear modulus in one specimen/sample of simple form. Using this method it is required either victuals or rations of filament to specimen/sample, there is also no necessity to establish/install in it tags for fastening of filaments. As a result the replacement of specimen/samples is accelerated and is simplified. The described method of the suspension of specimen/sample is used on installations UP-4, UP-5, UP-6.

The comparative tests of the methods of the suspension of specimen/sample (Fig. 25c, d, e) conducted showed that the method of the suspension of specimen/sample in the loops of filaments does not affect substantially resonance frequency.

The diagram of installation UP-4 is given to Fig. 26. Test specimen 1 is hung in the loops of filaments. As filaments is utilized copper and Nichrome wire with diameter 0.1 mm. Specimen/samples for determining Young's modulus and shear modulus are the rods with a diameter of 7-8 mm, by length 70-200 mm. Young's modulus can be determined also on bars. As exciter and receiver, are used sound-pickup heads ZPK-55 whose piezoelectric cell is made from ceramics of titanate of barium. The filament during which will hang the specimen/sample, is connected to the needle holder of head. The head is establish/installed in such position that during the supplying of variable stress on the piezoelectric cell of filament are imparted the longitudinal variables. Exciter and receiver of the oscillations are installed on a movable bracket, which makes it possible to change the distance between them along the length of test specimens. Electronic equipment, utilized for exciting of oscillations and recording of resonance frequency at installation UP-4, and also during the described below high-temperature installations, the same as during installation UP-1.

Exciter 3 feeds from the audiofrequency oscillator of 7 types ZG-12, of oscillations of specimen/sample they are absorbed by receiver 2, signal from which is strengthened by amplifier 4 and is supplied to oscilloscope 5, utilized as the marker of resonance. Coarse reading of frequency is produced on the dial/limb of generator

7, and precise - by electronic frequency meter 6.

Lowering bracket with exciter and receiver, specimen/sample can be placed into the working chamber of the furnace of resistance to 8 for determining of elasticity characteristics at elevated temperatures. The temperature of test specimen is checked by three thermocouples 10 on potentiometer 11. Thermocouples are attached to the middle also of the ends of test specimen/sample 9. Control specimen/sample is placed in working chamber of furnace 8 at a distance of 5 mm from test specimen. Exciter and receiver are cooled by air flow from fan.

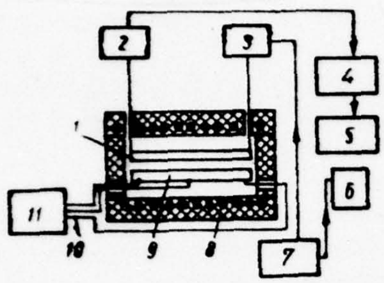

Fig. 26. Block diagram of installation UP-4.

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Installation UP-4 can be also used for determining of elasticity characteristics at the reduced temperatures. For this, the specimen/sample is placed into the cryostat, coolant in which is liquid nitrogen.

2. Determination of the moduli of elasticity at high temperatures.

The majorities of testing units for determining of elasticity characteristics in the heated specimen/sample which are described in the literature, created for a work in the range of temperatures from room to 1500° K. They are intended basically for tests of heat-resistant steels, nickel and cobalt alloys. To utilize them for determining of elasticity characteristics of high-melting materials turned out to be impossible as a result of the low temperature of

heating specimen/sample, so therefore high-melting materials possess a series of special feature/peculiarities, which do not make it possible to experience/test them on installations of such type. Will be required the development of special test procedure and the creation of such installations whose construction/design makes it possible to consider the specific character of high-melting materials.

It is known that the refractory metals (tungsten, molybdenum, niobium, etc.) strongly are oxidized during heating in air; therefore the high-temperature tests of these metals and their alloys can be carried out only in vacuum or in the atmosphere of inert gases of high frequency.

The number of high-melting materials, especially on the basis of refractory compounds, yield with difficulty or yield in no way to machining. Specimen/samples of them, manufactured with methods powder metallurgy, can be obtained only simple form, limited size/dimensions, moreover not always even round cross-section.

In view of the fact that some high-melting materials are in a small quantity, a number of manufactured of them specimen/samples and size/dimensions of the latter are limited. Many high-melting materials scatter to a considerable extent vibrational energy;

therefore the disturbance of oscillations in specimen/sample must be sufficient to intense ones. Furthermore, with an increase in the temperature the dissipation of energy grow/rises; therefore is required the application/use of more powerful vibration exciters and sensitive receivers. For these purposes are suitable electromagnetic, magnetostrictive and piezoelectric vibration exciters, but the first two types of exciters give considerable focusing/inductions to receiving sensors. To remove focusing/inductions via careful screening in the high-temperature vacuum camera/chamber is difficult.

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Important value has an ability of exciters and receivers to work at elevated temperatures. For the electromagnetic and magnetostrictive exciters the greatest temperature is the Curie point (loss of magnetic properties), while for piezoelectric ones - temperature of the disappearance of piezoelectric effect, which, for example, for Rochelle salt is lower than 400° K.

The application/use of electromagnetic, magnetostrictive and piezoelectric exciters and receivers during high-temperature tests is possible only if the excitation of oscillations in specimen/sample and their procedure from specimen/sample will be produced through some adapter —— rod or filament.

Exciter and receiver of oscillations must be sufficiently distant from high-temperature zone and equipped by additional cooling.

During high-temperature tests specimen/sample is heated either from external heat sources because of radiation or thermal conductivity or because of heat liberation within it alone. In the last case through the specimen/sample, is passed the electric current or is utilized the induction method of heating. Specimen/sample desirable to fasten so that the rod is with free ends. In view of the fact that the high-melting materials can be current-conducting and noncurrent-conducting, in this case most adequate/approaching is the radiation method of heating.

It is necessary to note also, that even so more easily in all to excite oscillations of specimen/sample, which is long and stem however in this specimen/sample is difficult to ensure uniform heating along the length. The account to the nonuniformity of heating specimen/sample, although it is feasible, is sufficiently bulky. When selecting of the size/dimensions of specimen/samples made of high-melting materials for high-temperature tests, one should take into consideration that with the decrease of the diameter of

specimen/sample grow/rises the role of the material of the surface layers which usually have a large quantity of different flaw/defects.

On the basis of that presented above, the most suitable can be considered the test samples with a diameter of 7-9 mm and with a length of 70-120 mm. During the high-temperature tests of high-melting materials for measuring the temperature of specimen/sample, are utilized the pyrometers and the thermocouples. Under conditions of the radiation heating of specimen/sample during the measurement of temperature by thermocouples are obtained more accurate results, than with measurement by its optical pyrometer. The optical pyrometers show real temperature only when the emission/radiation of the incandescent body whose temperature is subject to measurement, it is sufficiently close to blackbody radiation. Under other conditions into readings of pyrometer, it is necessary to introduce corrections.

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Applying the radiation heating of specimen/sample, it is difficult to avoid the incidence on them of the light rays of heater reflected into the objective of pyrometer, which increases the apparent brightness of specimen/sample and distorts readings of the optical pyrometer.

A shortcoming in the optical pyrometers is also the fact that they can measure the temperature of specimen/sample not on all temperature range from room to high, but only that which is higher than the temperature of the visible brightness.

Thermocouples do not have these shortcomings. In neutral medium or vacuum they can be applied without any protective clothing. For measurements at high temperatures, are utilized platinum-platinum-rhodium, tungsten-molybdenum thermocouples, and also thermocouples from tungsten fusions with rhenium. The characteristics of thermocouples are given to Fig. 27 [11].

During the prolonged application/use thermocouples of platinum-platinum-rhodium group, make it possible to measure the temperatures to 1600° K, while at short-term - to 1870° K. They have good stability of thermoelectric properties. The manufacture of the junction of platinum-platinum-rhodium thermocouples does not cause difficulties. A shortcoming in the platinum-platinum-rhodium thermocouples is the low level of the measured temperatures. Maximum emf, developed with thermocouple PR 10/0 at 1870° K, is equal to 16.766 mV (on GOST 3044-45).

Tungsten-molybdenum thermocouples it makes it possible to measure the temperatures to 2300° K. Of this best of the thermocouples group is thermocouple TsNIIChM-1 (tungsten-molybdenum from 0.5o/o aluminum). It develops considerably greater emf in comparison with others, but it has low stability.

Thermocouples of tungsten-rhenic alloys make it possible to measure the temperatures higher than 2500° K. These thermocouples develop high emf and possess good stability. The best of them is thermocouple VR5/20. This thermocouple is already thoroughly calibrated to the melting points of pure metals up to 3300° K [28].

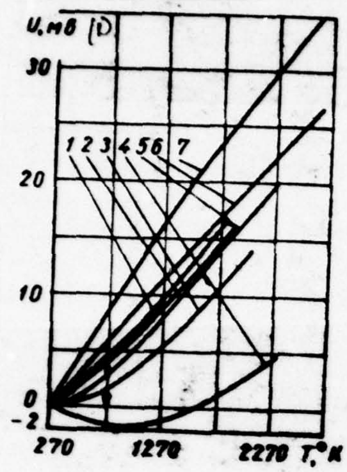

Fig. 27. The characteristics of the high-temperature thermocouples: 1 - PR10/0; 2 - PR13/1; 3 - PR30/6; 4 - tungsten-molybdenum; 5 - TsNIIChM-1; 6 - VR3/15; 7 - VR5/20.

Key: (1). mV.

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Parts and assemblies of the installations, intended for the tests of materials at temperatures are higher than 2300° K, they must be made from the materials which can reliably operate at such high temperatures. Table 1 [60] gives the metallic and nonmetallic materials, used at present for manufacturing the heaters, that work in different temperature intervals. In laboratory practice great application/use will obtain tungsten, molybdenum, tantalum and

graphite heaters [33]. Heaters from nonmetallic materials are common mainly in industrial installations.

During design of the heater and the selection of material is necessary to focus attention on operating temperatures, workability of material at normal temperature and its mechanical strength at high temperatures. From the materials indicated good machinability possesses the molybdenum; however, in it low strength and stability at high temperatures. The application/use of tantalum is limited mainly to its high cost/value.

Table 1. Materials for high-temperature devices.

(1) Материал	(2) Состав, %	(3) Максимальная рабочая температура для приемлемого срока службы, °К	(4) Рекомендуемая рабочая атмосфера
(5) Платина (проволока, лента)	Pt—100 (номинально) (6)	1670	(7) Воздух
Сплав платины и родия (проволока) (8)	Pt—87, Ph—13; Pt—90, Ph—10; Pt—60, Ph—40	1810	(7) Воздух
Молибден (проволока, полоса, пруток) (8а)	Mo—100 (номинально) (6)	1920—2470 При более высоких температурах сокращается срок службы (9)	Вакуум $5 \cdot 10^3$ *мм рт.ст.* водорода при атмосферном или пониженном давлении (10)
Молибден с защитным поверхностным слоем (пруток) (11)	Основа Mo—100 (номинально) Покрытие $MoSi_2$ (основная составляющая) (12)	270 1800—2070	(7) Воздух
Тантал (проволока, пруток, полоса) (13)	Ta—100 (номинально) (6)	2270	Вакуум (14)
Вольфрам (проволока, пруток, полоса) (15)	W—100 (номинально) (6)	1970—2770 При более высоких температурах сокращается срок службы (9)	Вакуум 10^4—10^5 *мм рт. ст.*, чистый водород (16)
Неметаллические материалы Глобар (прутки, трубы) (17)	SiC+связка (18)	(9) 1670—1870 При более высоких температурах сокращается продолжительность службы	(7) Воздух

Table 1. Continued

Дисилицид молибдена (пруток) (20)	$MoSi_2$	1970	Воздух (5)
Окислы или смесь окислов (прутки) (21)	ThO_2—96, (22) Y_2O_3 или L_2O_3—4, ThO_2—85, CeO_2—15	2220—2270 При температуре выше 1850 смесь с CeO_2 дает лучшие результаты, чем смесь с Y_2O_3 или La_2O_3 (23)	Воздух (5)
Угольные и графитовые материалы (прутки, трубы, спирали, вырезанные из труб) (24)	C—100 (номинально) (6)	2270—3270 При более высоких температурах сокращается срок службы (9)	Вакуум, нейтральная или восстановительная атмосфера (25)
Стабилизированная двуокись циркония (26)	ZrO_2—100 (номинально) (6)	2670	Воздух (5)

Key: (1). Material. (2). Composition. (3). Maximum operating temperature for acceptable service life. (4). Recommended working atmosphere. (5). Platinum (wire, strip). (6). nominally. (7). Air. (8). Alloy of platinum and rhodium (wire). (8a). Molybdenum (wire, band, rod). (9). At higher temperatures is shortened service life. (10). Vacuum $5 \cdot 10^3$ mm Hg of hydrogen with atmospheric or lowered pressure. (11). Molybdenum with shielding surface layer (rod). (12). Basis Mo-100 (nominally). Coating $MoSi_2$ (fundamental component). (13). Tantalum (wire, rod, band). (14). Vacuum. (15). Tungsten (wire, rod, band). (16). Vacuum 10^4-10^5 mm Hg, pure hydrogen. (17). Nonmetallic materials. Globar (bars, tubes). (18). binder. (19). At higher temperatures is shortened service life. (20). Disilicyl of molybdenum (rod). (21). Oxides or mixture of oxides (rods). (22). or. (23). At temperature above 1850 mixture with CeO_2 gives best results, than mixture with Y_2O_3 or La_2O_3. (24). Carbon and graphite materials (rods, tubes, helixes, cut out from tubes). (25). Vacuum, neutral or reducing atmosphere. (26). Stabilized dioxide of zirconium.

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The highest melting is tungsten, which possesses the same sufficient strength at high temperatures, but it is worked with difficulty at normal temperature. Therefore the heaters, manufactured from tungsten, must have the simple form, which allow/assumes the

treatment by abrasive tccl.

For applying the material at high temperatures, has also value of the vapor pressure and rate of evaporaticn in dependence on temperature. Are given below the maximum permissible values of the temperature by which the evaporaticn after 100 h does not exceed 1 weight o/o (these for a cube with side 1 cm during heating in high vacuum) [60].

(1) Вольфрам	2830	(8) Рутений	2170
(2) Тантал	2670	(9) Родий	1940
(3) Рений	2650	(10) Платина	1870
(4) Ниобий	2500	(11) Цирконий	1710
(5) Осмий	2380	(12) Титан	1380
(6) Иридий	2260	(13) Хром	1065
(7) Молибден	2180		

Key: (1). Tungsten. (2). Tantalum. (3). Rhenium. (4). Niobium. (5). Osmium. (6). Iridium. (7). Molybdenum. (8). Ruthenium. (9). Rhodium. (10). Platinum. (11). Zircorium. (12). Titanium. (13). Chromium.

From these data it is evident that for manufacturing of heaters most of all is suitable the tungsten.

Graphite as material fcr heaters together with positive quality (high operating temperature) has the shortccming: frcm it it is difficult to remove the absorbed by it gases. This leads to its gradual combustion, and the releasing products cf burning interact with the material of specimen/sample.

AD-A063 557 FOREIGN TECHNOLOGY DIV WRIGHT-PATTERSON AFB OHIO F/G 11/2
ELASTICITY CHARACTERISTICS OF MATERIALS AT HIGH TEMPERATURE,(U)
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The heat shields, installed around heater, usually are manufactured from the same materials, as heater. Are utilized plates from tungsten, molybdenum, tantalum with a thickness of 0.1-0.3 mm. Widest use received molybdenum shields. Are applied also shields from ceramic materials.

Different fasteners (screws, rivets, washer) most frequently are manufactured from molybdenum. High-melting electrical insulating materials are utilized for isolation/insulation of the cell/elements of heater one from another and from the surrounding parts, and also for isolation/insulation of the wires of thermocouples. For this purpose are applied mainly different high-melting oxides. The high-melting ones are oxides of thorium, magnesium and beryllium. The best electrical indices possesses oxide of beryllium and furthermore on strength at the temperature higher than 1870° K it exceeds all remaining oxides. However, oxide of beryllium is very toxic road. During the use of articles made of it, it is required to take the special precautionary measures against the incidence/impingement into the organism of its dust and vapors. High toxicity is the main reason being of limited usefullness of this oxide.

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The difficulties, which appear during the use of magnesium cxide, are connected with the fact that this oside is hydrated with contact with water. For preventing the dehydration, is required special high-temperature annealing.

Widest application will obtain alumina (Al_2O_3). This oxide is among chemically most stable and possesses high strength at temperatures to 2270° K. Main shortcoming in the alumina - comparatively low temperature of melting (2288° K). The manufacture of different articles made of alumina (crucibles, shielding covers for thermocouples, muffles) is is well familiar by industry.

Zirconium dioxide at room temperature is insulator, but at 2270° K its resistivity is less than 1 Ω•cm. Therefore it is unsuitable for use as electrical insulator at high temperatures. However, zirconium dioxide is a good material for heat shields because of refractoriness and low thermal conductivity.

The first of the develcped by us installations, intended for determining Young's modulus in the range of temperatures from room to 2000° K, will be installation UP-3 (Fig. 28) [21, 32]. Determination of Young's modulus on this installation is based on the measurements

of the resonance frequency cf the longitudinal oscillations of test sample.

Specimen/sample 1 is arranged vertically in the working space of the high-temperature camera/chamber. Its lcngitudinal oscillations are excited by magnetostriction oscillator 12, on ceramic tip of which the specimen/sample rests directly by lcwer end/face. In vertical position the specimen/sample is supported figure by plate 2. Plate is made from laminated molybdenum with a thickness of 0.1 mm and has a hole in which enters the upper part of the specimen/sample, concerning it at several points by side surface. The oscillations of specimen/sample are absorbed by rod 3, attached in rubber diaphragms 5, and they are transferred to piezoelectric receiver by 4. Signal frcm receiver is strengthened by amplifier 8 and enters the entry of oscilloscope 9. The windings of vibrator feed from master oscillator by 11. Changing smoothly oscillator frequency, is found this value, at which begin resonance cscillaticns of specimen/sample.

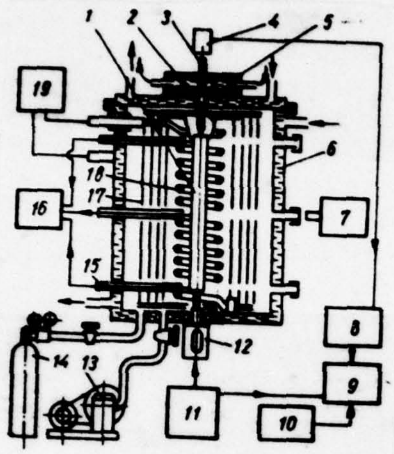

Fig. 28. Block diagram cf installation UP-3.

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Syntonization of sample is conducted according to the maximum of the amplitude of oscillations on the shield of cscilloscope 9. On the dial/limb of the master cscillator, is produced coarse reading of frequency. The precise measurement of frequency obtains during the comparison of the frequency of the driving oscillator with the frequency of heterodyne wavemeter 10 on Lissajcts figures on the shield of electron oscillcgraph or electronic frequency meter.

Specimen/sample heats in inert medium by heater 18. Air from the camera/chamber is evacuated by fore pump 13, and then it is filled with inert gas from bottle 14. Pressure in the camera/chamber during

tests is kept constant. The working space of the camera/chamber is heat-insulated by shields 17.

Temperature is measured in the middle of specimen/sample and in its end/faces by thermocouples 15, and also by pyrometer 7. High-temperature camera/chamber 6 is mounted on laboratory bench, and together on strut is arrange/located entire/all utilized equipment.

Is below given the more detailed description of some most important assemblies of installation UP-3 and of utilized specimen/sample.

Vibration exciter. Figure 29 shows the construction/design of vibration exciter and the diagram of its fastening to the bottom of the camera/chamber. Two-core magnetostrictor 1 is rigidly welded to adapter 2. In the end/face of adapter, is inserted tip by 3 of high-melting ceramics. Magnetostrictor is collected from the nickel plates with a thickness of 0.1 mm. Natural frequency of its longitudinal oscillations about 30 kHz. The size/dimensions of the plates of magnetostrictor are designed from equation [59]

$$\operatorname{tg} ka \operatorname{tg} kb = \frac{m}{n}, \qquad (108)$$

where $k=\frac{2\pi f_{np}}{c}$ (f_{np} —the natural frequency of the longitudinal oscillations of the plate: c - velocity of propagation of

longitudinal elastic wave in nickel rod, equal to $4.76 \cdot 10^5$ to cm/s).

The plates of magnetostrictor are manufactured with milling. Then they are cleaned and are cemented by glue BF into the bundle whose area comprises approximately 20x20 mm. The width of the rods of magnetostrictor will be approximately one half of the width of its window ($m \approx 0.5\ n$), with this $b = m$. The window of magnetostrictor is completely filled with the winding from wire/conductor in thickness approximately 0.15 mm. Adapter was made made of heat-resistant steel in the form of cylinder with the square base whose area was equal to the area of the end/face of vibrator. For the reduction of the weight of adapter and heat emission from specimen/sample to vibrator in the cylindrical part of the adapter, is made the drilling from the side of vibrator so that the walls of cylinder have a thickness of approximately 1 mm. Ceramic rod 3 (Fig. 29), inserted into deepening on the end/face of adapter, prevents the superheating of the latter.

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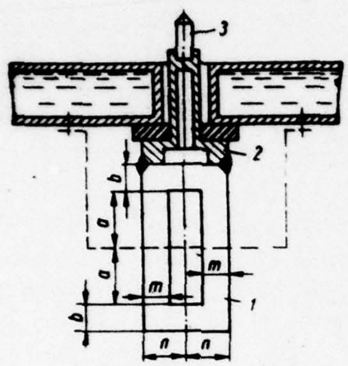
The diagram of fastening vibration exciter to housing is shown on Fig. 29 by dashed line. In junction/unit of the plane the magnetostrictor is pressed by the clamp, drawn to the bottom of the camera/chamber. The seal of joint is reached with the aid of rubber packing between adapter and bottom of the camera/chamber.

The excitation of the oscillations of specimen/sample is provided not only in the region of the natural frequency of magnetostrictor, but also over a wide range of frequencies (during installation they conducted measurements in the interval of 15-50 kHz). Magnetostrictor effectively works without magnetic biasing.

Receiver of oscillations. The oscillations of specimen/sample are recorded by special device (Fig. 30). Sensing element is plate 1 of polarized ceramics of titanate of barium. It is glued to flat spring 2, attached cantilever on a special bracket. Bracket makes it possible to move spring with plate in different directions. In operating position the plate of titanate of barium is pressed by the small force to the point of the rod, through which are transferred the oscillations from specimen/sample. This rod is made compound/composite: its upper part 3 - copper, and lower 5 - molybdenum. This construction/design of rod contributes to its effective cooling. The copper part of the rod is pressed between rubber diaphragms 4 and washes by running water.

Heater. Heater is helix from tungsten or molybdenum wire with a diameter of 1.0-1.5 mm. The turns of helix are isolate/insulated one from another and from adjacent parts by beads of oxide of aluminum.

For decreasing the nonuniformity of heating specimen/sample along the length, the heater drunken of the winding cn: less frequent in center and it is thicker on edges.



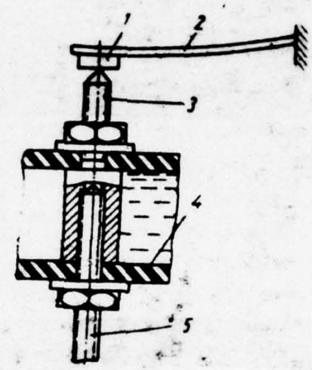


Fig. 29.

Fig. 30.

Fig. 29. Vibration exciter in installation UP-3.

Fig. 30. Receiver of oscillations in installaticn UP-3.

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Heater feeds from autotransformer 19 (see Fig. 28) type RNO-10. The butt end of the heater and one of the terminals of autotransformer are connected to the housing of the high-temperature camera/chamber. The heater of this construction/design makes it possible to heat specimen/sample to 2000°K, which limits the application/use of oxide of aluminum for isolation/insulation of its turns.

Shields. The thermal insulation of the working space of the camera/chamber is realize/accomplished by vertical and horizontal shields from molybdenum and nickel tin. Vertical shields are the cylinders, distant one from another by 3-4 mm. Several first shields from the side of heater - molybdenum, and others - nickel. Bottom and cap/cover of the camera/chamber also have screening. In shields are made the holes for the supply of thermocouples and measurement of the temperature of specimen/sample by a pyrometer.

Thermocouples. During installation UP-3, are utilized tungsten-molybdenum thermocouples VNIIcermet-1 and tungsten-rhenium VR5/20. Thermocouple wires are reinforced with a stick made of oxide

cf aluminum. For measurement by emf of thermocouples, was utilized the potentiometer of direct current 16 (see Fig. 28) type PP or electronic potentiometer EPP-09. The thermocouple junctions concern the surface of specimen/sample. The presence of the contact between the specimen/sample and the thermocouple is checked on the closing/shorting of low-current monitoring circuit.

Housing of the high-temperature camera/chamber. Housing is cylinder with double walls and bottom. On top it is closed by removable cover. The seal of the camera/chamber is achieved by rubber gasket. Housing and cap/cover of the camera/chamber are cooled by running water. In chamber casing, there are holes for the input/introduction of thermocouples, window, which make it possible to measure the temperature of specimen/sample with pyrometer, branch for the evacuation of air and filling of the camera/chamber with inert gas.

Specimen/sample. Specimen/sample is the cylindrical or prismatic rod with a length of 90 mm and by the transverse size/dimensions of 6-9 mm. In the end/faces of specimen/sample along its axle/axis, there are insignificant deepenings, where enter the points of the ceramic tip of vibrator and molybdenum rod of the receiver of oscillations, thanks to which the specimen/sample is supported in vertical position.

The determination of Young's modulus during installation UP-3 has a series of special feature/peculiarities. First of all specimen/sample, which is located in the high-temperature camera/chamber, one must not consider rod with free ends. By lower end/face it rests on the tip of vibrator, its upper end/face concerns receiving pin, and the lateral surfaces match up thermocouple wires. The factors indicated affect the resonance frequency of specimen/sample.

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Consequently, during the calculation of Young's modulus according to the expression, obtained for a rod with free ends, this effect must be considered.

The analytical account of the conditions of attachment in this case is very difficult; therefore the preliminarily fundamental resonance frequency of specimen/sample is determined during installation UP-1 at the normal temperature where the specimen/sample is located under conditions, similar to those under which is the rod with free ends. After this the specimen/sample is establish/installed for installation UP-3 and again is determined its resonance

frequency. The obtained difference in frequencies (usually $\Delta f \approx 0.003f$) is accepted for constant correction during the measurements of resonance frequency for the heated specimen/sample. Such measurements are produced in each specimen/sample. At the same time it is necessary to consider the nonuniformity of heating specimen/sample, since, in spite of the accepted measures, the temperature of the end/faces of specimen/sample will usually be below the temperature of its middle part by 5-10c/c.

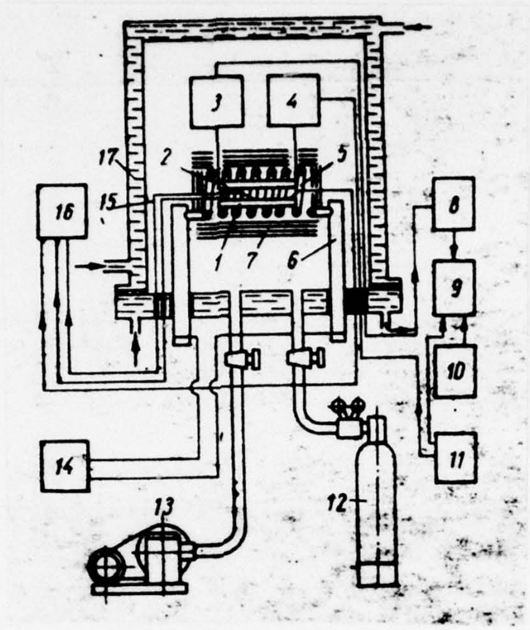

Fig. 31. The block diagram cf installation UP-5: 1 - control specimen/sample; 2 - test specimen; 3 - detector of oscillations; 4 - vibration exciter; 5 - heater; 6 - current supply; 7 - shields; 8 - amplifier; 9 - cathode cscilloscope; 10 - frequency meter; 11 - master oscillator; 12 - bottle with inert gas; 13 - forevacuum pump; 14 - variator; 15 - thermocouple; 16 - potentiometer; 17 - chamber casing.

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For determining Young's modulus and displacement in one specimen/sample at the high temperatures in inert medium we have

developed installation UP-5 (Fig. 31). Measurements on it are produced just as during installaticn UP-4; elastic modulus it is determined from the rescnance frequency of transverse vibrations, and shear modulus - in the resonance frequency of torsional oscillations. The complex of equipment, used for exciting oscillations and recording the resonance frequency in both installations is identical.

The test specimen with a length of 70-90 mm and with a diameter of 7-8 mm is hung in the locps of filaments in the working space of the high-temperature camera/chamber. By filaments serves tungsten and molybdenum wire with a diameter of 0.3 mm.

The temperature of specimen/sample is determined by pyrometer and thermocouples 15 in middle and in its end/faces. Since to attach thermocouples to test specimen is not impossible (they do not make it possible to excite the fluctuation of the specimen/sample of sufficient intensity), by thermocouples is measured the temperature of control specimen/sample 1, which is located under subject at a distance of 5 mm. However, by special investigations it is establish/installed, that during heating of subject and control room of specimen/samples with the thermocouples, attached to both specimen/samples, their temperature is identical. Readings of thermocouples are recorded by 16 types electronic potentiometer EPP-09 or by potentiometer of a direct current of the type PP. The

separate assemblies of installation UP-5 possess a series of special feature/peculiarities in connection with their use at high temperatures. Their detailed description is given below.

Exciter and receiver of oscillations (Fig. 32). By exciter 1 and as the receiver of 2 oscillations serve the sound pickup heads ZPK-55M. They are placed in water-cooled housings by 3. The piezoelectric cells of the sound pickup heads have additional cooling.

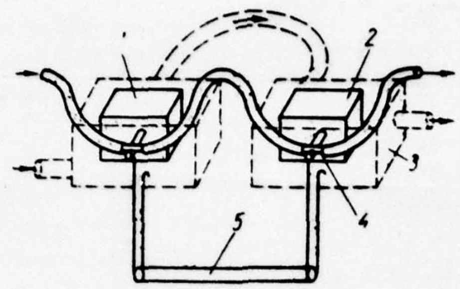

Fig. 32. Exciter and receiver of oscillaticrs.

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For this, to the holder of the piezoelectric cell, to which are hung the ends of the filaments, connected with specimen/sample 5 is connected tube with 4, thrcugh which is passed the water. The housing cf exciter and receiver of cscillations is attached on the mobile bracket which makes it possible to change the distance between filaments, to build up and to lower them.

Heater. With installation UP-5 can be utilized the heaters of two construction/designs. Heater from tungsten cr molybdenum wire is simple on device and does nct require special transformer. It is the helix whose turns are isclate/insulated frcm each other by beads from oxide of aluminum. Helix drunken of winding on - it is less frequent on middle and it is thicker on edges - for providing the uniform heating of specimen/sample. Heater is fastened with its

branch/removals to the copper water-cooled current supplies. This heater makes it possible to carry out tests at temperature to 2000°K. This constraint is associated with used on it insulating ceramics.

During heating to the temperatures higher than 2000°K, is utilized the strip heater (Fig. 33). Four plates 5 of laminated tungsten with a thickness of 0.3-0.5 mm are furnished horizontally. They are fastened with their ends to water-cooled supports 3 and 7. For the compensation for the elongation of tungsten plates during heating, left support 7 is made mobile and is drawn down by spring 6 of the tungsten wire. Heater feeds from transformer OSU-40, controlled by variator RNO-10. Test specimen 1 is hung into space between plates. The temperature of specimen/sample is measured by the pyrometer through the slot between the plates of heater, and it is also determined by measurement by thermocouples 4 of the temperature of control specimen/sample 2, which is located at a distance of 5 mm from subject.

Shields. The thermal insulation of the working space of the camera/chamber is realize/accomplished by shields from molybdenum tin. The upper part of the shields is removable. This makes it possible to build up and to lower specimen/sample into the working space of heater.

High-temperature camera/chamber. The high-temperature camera/chamber consists of the water-cooled plate/slab, which is covered on top with the cap/hood, also water-cooled. In cap/hood there are three inspection window for observation and measuring the temperature of test specimen by pyrometer.

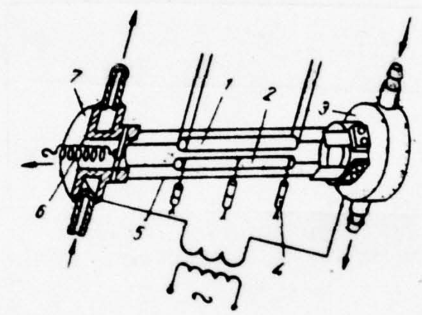

Fig. 33. The strip heater.

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Through the plate/slab pass current supplies to heater, branches those supplying water for cooling of exciter and receiver of oscillations, evacuation of air, supply of inert gas, conclusion/derivation of the wire/conductors of thermocouples. The high-temperature camera/chamber is mounted on separate stand. The forevacuum pump and bottle with inert gas are placed near panel.

The determination of Young's modulus during installation UP-5 differs in the fact that during this installation are investigated so-called short test samples ($l/d \approx 10$). The calculation of Young's modulus according to expression (73), in which are not considered the effects of displacement and rotation of the cell/elements of rod, it does not make it possible to obtain its precise value. The analytical methods the account of this value are given in chapter II; however, they require conducting complex mathematical computations. Therefore for each specimen/sample Young's modulus is preliminarily determined by us during installation UP-1 at normal temperature. Then specimen/sample is establish/installed for the installation UP-5, is

determined the resonance frequency of its transverse vibrations and Young's modulus they design from expression (73). The difference in values accepts as constant correction to the values of Young's modulus, obtained at high temperatures.

Experiment showed that the installaticn cf this construction/design as UP-5, is most appropriate for determining of elasticity characteristics at high temperatures. The used diagram of the suspension of specimen/sample during fire/thin filaments makes it possible to remove exciter and receiver of the cscillations of specimen/sample which fail the high temperatures, from the heating zone, but specimen/sample tc place in the werking space of heater. In this diagram it is possible to determine Young's modulus and shear modulus simultaneously in one specimen/sample, which is also important, since some new materials are even in a small quantity. Furthermore, with obtaining of values of E and G in one specimen/sample it is pcssible to more accurately calculate Poisson ratio for a tested material.

On the basis of the above for determining cf elasticity characteristics of high-melting materials at the high temperatures in vacuum was developed the installation UP-6 ¹ (Fig. 34) [15], the operating principle of which the same as installation UP-5.

FOOTNOTE [1]. Development, adjustment and operation of installation UP-6 are carried out by the author together with V. A. Dreshpak. ENDFOOTNOTE.

The construction/design of the most important assemblies of the high-temperature camera/chamber of installation UP-6 is more advanced, and those of them, that are subjected high temperature effect, they have effective cooling. Therefore during installation UP-6, it is possible to determine elasticity characteristics at temperature to 3300°K.

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Installation consists of the high-temperature vacuum camera/chamber, the equipment for the creation of vacuum and heating of specimen/sample, complex of equipment for exciting the oscillations of specimen/sample and recording of its resonance frequency, measurement of the temperature of specimen/sample, checking of evacuation/rarefaction in the camera/chamber, and also different signal and protecting devices.

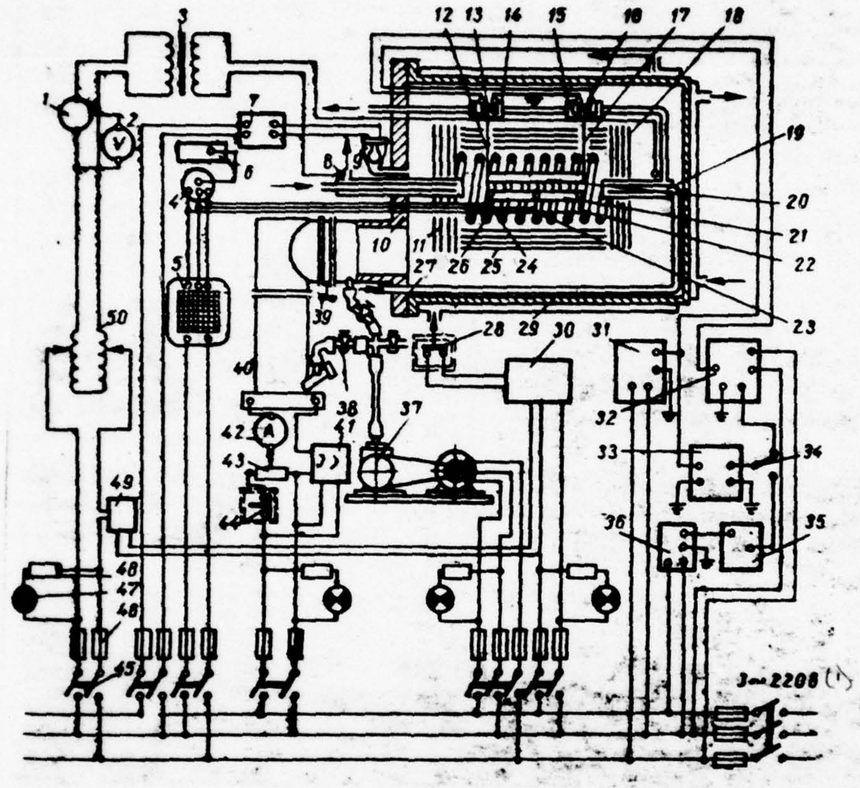

Fig. 34. Block diagram of installation UP-6. 1 - ammeter of alternating current; 2 - voltmeter; 3 - transformer of the type CSU-40/05; 4 - switch of thermocouples; 5 - electronic potentiometer of the type EPP-09; 6 - potentiometer of the type PP; 7 - vacuum gauge of type ~~twisted-1;~~ VIT-1; 8 - fixed current conductor; 9 - vacuum gauge tubes LT-2 and LM-2; 10 - plate/slab; 11, 18 - vertical shields; 12, 17 - wire suspensions; 13 - receiver of oscillations; 14, 15 - holders of exciter and receiver; 16 - vibration exciter; 19 - mobile conductor; 20 - test specimen; 21 - control specimen/sample; 22-24 -

thermocouple; 25 - horizontal shields; 26 - heater; 27 - packing layer; 28, 44 - relay of water pressure; 29 - cap/hood; 30 - relay MKU-48; 31 - master oscillator; 32 - amplifier; 33 - the cathode-ray oscilloscope; 34 - single-pole switch; 35 - scaler; 36 - heterodyne wavemeter; 37 - fore pump; 38 - vacuum tap/crane; 39 - vacuum catch; 40 - diffusion pump; 41 - overload relay; 42 - ammeter; 43 - autotransformer; 45 - double-pole switch; 46 - predoxraniteli; 47 - signal tube; 48 - resistance; 49 - contactor; 50 - variator BOT-25/10.

Key: (1). V.

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The high-temperature vacuum camera/chamber (Fig. 35) consists of vertical plate/slab 1 and cylindrical cap/hood 2, arrange/located is horizontal. Cap/hood can be abstract/removed on guides, which provides free access to all internal assemblies of the camera/chamber and light/lung replacement of specimen/samples. By most adequate/approaching are the cylindrical specimen/samples with a diameter of 6-8 mm and with a length of 180-120 mm. Test specimen by 11 is hung during filaments in the working space of heater. Upper ends of threads are fastened to exciter with 3 and receiver of 4 oscillations which are establish/installed on water-cooled stand 5.

The devices of the vibration exciter and receiver are similar (Fig. 36). Driver is plate 1 of piezoceramics. This plate is attached by glass filament to holder 2. One of the conclusion/derivations of plate is grounded, and another conclusion/derivation matches up wire/conductor from the master oscillator or amplifier. Holder at one end has hook 4, to which is hung up the filament, which supports specimen/sample. The opposite end of the holder is soldered to tube with 3. The passing on tube water cools holder, without making it possible thus to be superheated for piezoceramic plate.

As heater serve four tungsten plates with a thickness of 0.5 mm which with its ends are fastened to the molybdenum tips of 10 copper current supplies 9 and 14 (see Fig. 35).

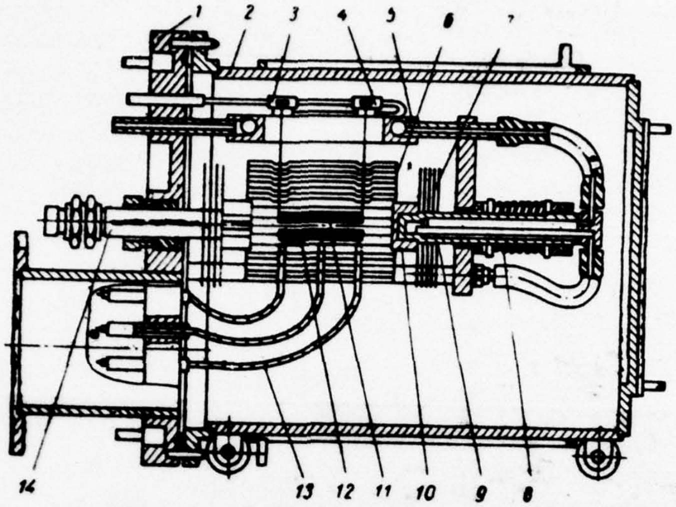

Fig. 35. High-temperature vacuum camera/chamber of installation UP-6.

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Right current supply 9 is made mobile. During the heating when the plates of heater are lengthened, it is drawn down by spring 8.

The working space of heater is encircled by vertical 7 and horizontal 6 shields, manufactured from tungsten, molybdenum and nickel tin with a thickness of 0.2-0.3 mm. Horizontal shields are the convolute into cylinder plates, which are located one from another at a distance of 3-4 mm. Vertical shields are assembled from the flat sheets, fastened between themselves screw/propellers, by spacers and nuts from tungsten and molybdenum. Nearest to heater plates tungsten,

remaining molybdenum and nickel.

Horizontal shields have removable forward section for the facilitation of the installation of specimen/samples. In them there are holes for the supply of thermocouples 13 (see Fig. 35) to control specimen/sample 12 and three windows for the measurement of the temperature of test specimen 11 by pyrometer. The corresponding windows with quartz glass are in the cap/hood of the camera/chamber.

For determining the temperature of test specimen, there is three tungsten-rhenic thermocouples of the mark/brand VR5/20. The thermocouple junctions are attached to middle and end/faces of the control specimen/sample, arrange/located under subject. To the part of the thermocouple wire, passing through shields, are put on the beads from oxide of aluminum.

During the work of the setting up of the wall of the high-temperature camera/chamber, the stand, on which are establish/installed the exciter and the receiver of oscillations, and also current supplies, intensely they are cooled by running water. Vacuum in the camera/chamber is created with the aid of fore pump VN-2 and diffusion pump N5S. Checking of evacuation/rarefaction is conducted with the aid of vacuum gauge VIT-1.

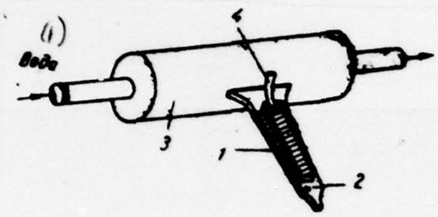

Fig. 36.

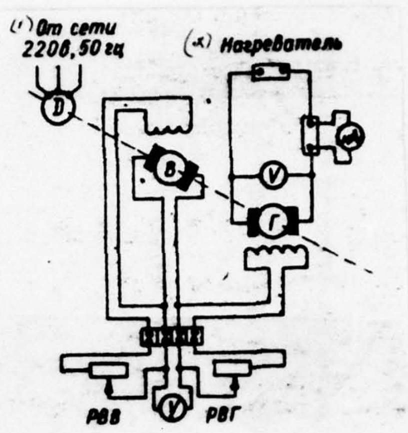

Fig. 37.

Fig. 36. Device of exciter and receiver of oscillations.

Key; (1). Water.

Fig. 37. Diagram of feeding of heater during setting up UP-6 by direct current.

Key: (1). From net 220 V, 50 Hz. (2). Heater.

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The feeding of heater is realize/accomplished from the industrial net of alternating current by voltage 220 V through the gauging

transformer ROT-25/10 and step-down transformer OSU-40/0.5. During the operation of setting up, noticed that in the case of heating specimen/sample it is higher than 2000°K, when on current supplies it passes the current of the significant magnitude, different circular parts, which encompass current supplies (nuts, washers, etc.), are heated by vortex currents. They take the place also of focusing/induction to wire/conductors from receiver to amplifier. In connection with this was developed the diagram of the feeding of heater by direct current (Fig. 37).

By the source of direct current serves motor generator AD5000/2500 in power 30 kW. Output potential its smoothly is regulated from zero to 6 or 12 V with the aid of two rheostats, connected in the circuit of the excitation windings of exciter and generator (RVV and RVG). The conditions/mode of heating is checked by the voltmeter and the kiloammeter, connected in the circuit of heater.

The use of heating by a direct current will make it possible to get rid of undesirable phenomena indicated above.

The general view of setting up UP-6 is shown on Fig. 38.

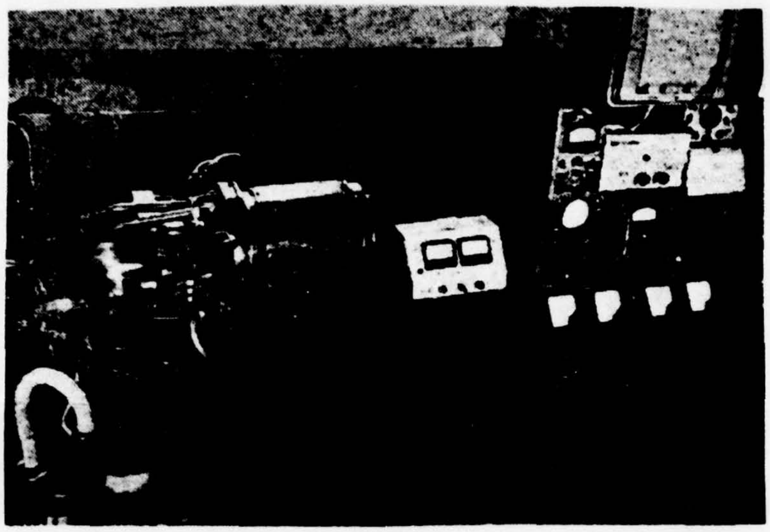

Fig. 38. The general view of setting up UP-6.

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3. Error analysis of determining elasticity characteristics during resonance settings up.

The accuracy/precision of determining elasticity characteristics during resonance settings up depends on the accuracy of the measurement of the values, entering the calculated dependences, and from accomplishing of those assumptions which were accepted during the derivation of these calculated dependences.

Let us estimate at first the errors, which occur during determining of elasticity characteristics.

With the determination of Young's modulus in the resonance frequency of longitudinal oscillations according to expression (52) a maximum relative error of measurement E will be

$$\frac{\Delta E}{E}=2\frac{\Delta l}{l}+\frac{\Delta\gamma}{\gamma}+2\frac{\Delta f}{f_{np}}, \qquad (109)$$

where $\frac{\Delta l}{l}$; $\frac{\Delta f}{f_{np}}$; $\frac{\Delta\gamma}{\gamma}$ - individual maximum relative errors in values l, f_{np}, γ.

The length of specimen/samples is measured usually by vernier caliper with an accuracy to 0.05 mm. Specific gravity/weight γ is determined calculation - from the weight of specimen/sample, its transverse size/dimensions and length. Specimen/sample they weigh on analytical balance with an accuracy to 0.01 g, and its transverse size/dimensions are measured by micrometer gauge with an accuracy to 0.01 mm. In that case, since

$$\gamma=\frac{4Q}{\pi d^2 l}, \qquad (110)$$

(Q - weight of specimen/sample), we have

$$\frac{\Delta\gamma}{\gamma}=\frac{\Delta Q}{Q}+2\frac{\Delta d}{d}+\frac{\Delta l}{l}. \qquad (111)$$

The accuracy of the measurement of the resonance frequency of specimen/sample depends on the accuracy/precision of the utilized frequency meter, and also on the character of the attachment of specimen/sample at node, the type of used of exciter and receiver of

oscillations, their interactions with specimen/sample.

With setting up UP-1 as instrument for frequency, measurement is applied electronic frequency meter 43-3, that has a maximum error of measurement 0.1o/o.

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Fastening specimen/sample in nodal section in the rubber blanket, and also excitation and reception of the oscillations of the specimen/sample through a layer of oil between converters and end/faces of specimen/sample give the possibility to find resonance frequency with maximum relative error 0.3o/c. Individual relative errors compose $\Delta l/l$=0.06o/o and $\Delta\gamma/\gamma$=0.35o/c. With such maximum individual relative errors a maximum relative error in the determination of Young's modulus during setting up UP-1 will be $\Delta E/E$=1o/o.

In the resonance frequency of transverse vibrations according to expression (73) Young's modulus is found with this maximum relative error:

$$\frac{\Delta E}{E}=4\frac{\Delta l}{l}+2\frac{\Delta d}{d}+\frac{\Delta\gamma}{\gamma}+2\frac{\Delta f}{f_{nn}}. \qquad (112)$$

Maximum relative errors of measurement of length, diameter and

specific gravity/weight of specimen/sample we have determined above. It is necessary to explain the accuracy of the measurement of resonance frequency. Since with settings UP-4, UP-5 and UP-6 effect from through the filaments from which is suspend/hung the specimen/sample, therefore, thickness, length and material of filaments, and also their arrangement will affect resonance frequency. It is important to explain the degree of this effect, since depending on the temperatures by which are produced the tests, for the suspension of specimen/sample can be used the filaments from different materials (copper, nichrome, molybdenum, tungsten), the thickness and the length of filaments can be different ones.

We have investigated the effect of the factors indicated on resonance frequency. Experiments are conducted during specimen/sample made of steel 45 and suspensions made of copper, tungsten and molybdenum.

Table 2. Resonance frequencies of specimen/samples (kHz) made of different materials witl different thickness of the filaments of suspension.

(1) Материал	d, мм	(2) Колебания	
		(3) поперечные	(4) крутильные
(5) Медь	0,1	3,602	15,863
(6) То же	0,3	3,579	15,858
(7) Молибден	0,3	3,547	15,843
(8) Вольфрам	0,3	3,538	15,844

Key: (1). Material. (2). Oscillaticns. (3). transverse. (4). torsion. (5). Copper. (6). The same. (7). Mclybdenum. (8). Tungsten.

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During filaments of one material of identical thickness, the resonance frequency of transverse and torsicnal oscillations is measured five times, and its mean value is introduced into table (Table 2). From the results of measurements, it is evident that with an increase in the thread diameter insignificantly is decreased the frequency and increases the scatter of the values of various measurements. With the suspension cf specimen/sample on molybdenum and tungsten filaments, the measured resonance frequency is lower than on copper ones. Thickness and material of filaments more greatly affect the resonance frequency of transverse vibrations, than torsion

ones. K. A. Bessonov and O. P. Stankevich [9], that investigated the effect of the copper suspensions of different diameter on the resonance frequency of transverse vibrations of brass specimen/sample, also will arrive at a similar conclusion.

In specimen/sample made of steel 45 we have investigated another effect of the length of the filaments of suspensions on resonance frequency. The length of suspensions is selected to the proportional length of longitudinal waves in filament at frequencies corresponding to the resonance frequencies of transverse and torsional oscillations. It was establish/installed, that the length of filaments does not substantially affect the resonance frequencies of transverse and torsional oscillations. The same results were obtained independent of us by B. A. Ovsyannikov, Ye. A. Kurganov and D. V. Lebedev [42].

In literature are contradictory data on the effect of the arrangement of suspension points on the resonance frequency of specimen/sample.

Table 3. The resonance frequencies of the oscillations of specimen/sample (kHz) with different arrangement of the filaments of suspension.

(1) Колебания	(2) Расстояние от торца образца, мм												
	1	2	3	4	6	8	10	12	14	16	18	20	22[1]
(3) Поперечные	3,601	3,603	3,602	3,603	3,604	3,603	3,603	3,603	3,602	3,602	3,603	3,602	3,602
(4) Крутильные	15,863	15,869	15,869	15,864	15,859	15,859	15,870	15,872	15,869	15,862	15,871	15,865	15,963

Key: (1). Oscillations. (2). Distance from end/face of specimen/sample, mm. (3). Transverse. (4). Torsion.

FOOTNOTE 1. The assembly of transverse vibrations is located at a distance of 22 mm from end/face. ENDFOOTNOTE.

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Thus, A. S. Matveyev, Ye. Kh. Bipp and L. S. Freyman [39] confirm that a change in the distance between suspensions does not affect the resonance frequency of specimen/sample, but in work [9] is made opposite conclusion.

We have measured the resonance frequencies of transverse and torsional oscillations during steel specimen/sample and copper

suspensions d=0.1 mm in different positions of suspension points - from the end/face of specimen/sample to the assembly of transverse vibrations. The obtained results (Table 3) show that the resonance frequencies, measured in different positions of suspension points, have deviations, which lie at limits of accuracy of measurements. Thus, there are no foundations for strictly standardizing distance from end/face to suspension point.

Let us assume that the resonance frequency of transverse vibrations is determined with maximum relative error by 0.3o/o, and let us take the values of individual relative errors, found earlier, then we will obtain that a maximum relative error in the determination of Young's modulus is equal to 1.2o/o.

During the determination of shear modulus from the resonance frequency of torsional oscillations (according to expression (55)), a maximum relative error in determination G is expressed by the formula

$$\frac{\Delta G}{G}=2\frac{\Delta l}{l}+\frac{\Delta\gamma}{\gamma}+2\frac{\Delta f}{f_{\text{кр}}}. \qquad (113)$$

Expression (113) completely coincides with expression (110), what is the consequence of the similarity of calculated dependences (55) and (52). In that case, with the obtained above maximum individual relative errors, a maximum relative error in the determination of shear modulus is equal to 1o/o.

For determining the maximum relative error in the Poisson ratio, let us record expression (4) in the form

$$\mu = 0{,}5\,EG^{-1} - 1. \qquad (114)$$

Since μ is function E and G, let us determine first a maximum absolute error in this function:

$$\Delta\mu = 0{,}5\,G^{-1}\Delta E + 0{,}5\,EG^{-2}\Delta G. \qquad (115)$$

Converting, we obtain

$$\Delta\mu = 0{,}5\,EG^{-1}\left(\frac{\Delta E}{E} + \frac{\Delta G}{G}\right). \qquad (116)$$

Then a maximum relative error in the determination of Poisson ratio

$$\frac{\Delta\mu}{\mu} = \frac{0{,}5\,G^{-1}\left(\frac{\Delta E}{E} + \frac{\Delta G}{G}\right)}{0{,}5\,EG^{-1} - 1}. \qquad (117)$$

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From expression (117) it follows that the accuracy/precision of the determination of Poisson ratio depends on the accuracy/precision of determination E and G. For example, if the values of Young's modulus and shear modulus, measured with maximum relative error 2.5o/o, are satisfactory ones, then during the determination of Poisson ratio maximum relative error reaches 24o/o, which is completely inadmissible. If Poisson ratio is designed from the values of the module/moduli of elasticity E and G, measured with maximum relative error 1o/o, then a maximum relative error in the

determination μ (for molybdenum) is equal tc 8o/o.

Let us pass to estimation of error, connected with accomplishing cf assumptions, accepted during the derivation of calculated dependences.

Expression (52) for determining the modulus of elasticity from the resonance frequency of the longitudinal oscillations of specimen/sample does not consider the inertia of the transverse motion of the parts of the rod. By Rayleigh is derived correction to account for this phenomenon. For a rod with free ends, the effect of the inertia of transverse motion consists of an increase in the period of oscillations of the first tone in the following sense:

$$1:\left(1+\frac{\mu^2\pi^2 r^2}{4l^2}\right), \tag{118}$$

where r - a radius of cross section.

Taking into account the correction of Rayleigh, we obtain expression for determining Young's modulus from the resonance frequency of longitudinal oscillations in the form

$$E=\frac{4l^2\gamma}{g}f_{np}\left(1+\frac{\mu^2\pi^2 r^2}{4l^2}\right) \tag{119}$$

It is necessary to note, however, that the values of Young's modulus, calculated taking the correction into account and without its account, differ insignificantly. So, on the data of work [31],

for the steel rod whose length 10 times greater than diameter, difference in values E does not exceed 0.1c/c.

During the derivation of expression (73) for determining Young's modulus from the resonance frequency of transverse vibrations is taken into attention only forward motion of cell/element dx, both rotary inertia and its displacement they disregard. This expression makes it possible to obtain a precise value of Young's modulus only in long specimen/samples. However, in connection with the fact that experience/testing specimen/samples at high temperatures, on a whole series of reasons they approach that so that the specimen/samples would be as shorter as possible, it is necessary to know, in what ratios l/d of the specimen/sample of the deviation of the values of Young's modulus from actual value they become considerable in order to use more precise calculated relationship/ratios.

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Such investigations were carried out by V. A. Kuz'menko [30] in the steel specimen/samples which have l/d=2.74-22.11. Beginning with setting up UP-1 at all specimen/samples was measured the resonance frequency of longitudinal oscillations and according to expression (52) is calculated the value of Young's modulus. As it was shown above, the value of Young's modulus, determined according to the

resonance frequency of the longitudinal oscillations of test sample, barely depends on the ratio l/d, in consequence of which this value of Young's modulus was accepted as real. Then during setting up UP-4 at all specimen/samples was measured the resonance frequency of transverse vibrations and according to expression (73) is calculated the value of Young's modulus.

In Fig. 39 curve 1 shows the difference in the percentages between the values of Young's modulus, obtained in one and the same specimen/samples in the resonance frequency of longitudinal and transverse vibrations. Since specimen/samples had different ratios l/d, then in essence curve 1 shows a change in the relative error in Young's modulus during his calculation in expression (73) depending on ratio l/d. Curve 1 visually shows that for the specimen/samples, which have l/d=10-15 which frequently are utilized in practice, a relative error in the determination of Young's modulus is very considerable (2-5o/o).

Work [30] shows also how to calculate Young's modulus the resonance frequency of transverse vibrations taking into account the rotary inertia and displacement. For obtaining V. A. Kuyz'menko's calculated relationship/ratios, proposes to utilize a simpler differential equation, than that that is given in S. P. Timoshenko's book [61] and in other sources. The equation, V. A. Kuz'menko's

proposed, takes the form

$$n^2 \frac{\partial^4 y}{\partial x^4} - \omega r_u \frac{\partial^4 y}{\partial x^2 \partial t^2} + \frac{\partial^2 y}{\partial t^2} = 0. \qquad (120)$$

Here

$$n^2 = \frac{EI}{\rho S} = r_u^2 \frac{E}{\rho}; \quad \omega = 1 + \frac{2}{h}(1 + \mu).$$

where h - coefficient, depending on the form of cross section.

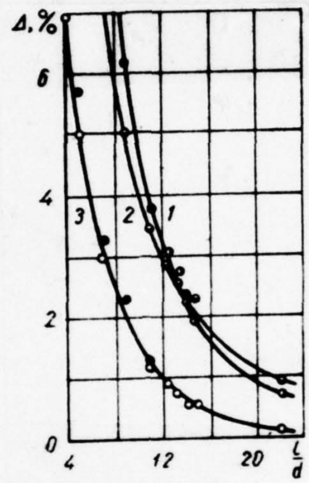

Fig. 39. Dependence of magnitude of error in the determination of Young's modulus from ratio l/d.

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The frequency equation, which corresponds to expression (120), will be

$$\operatorname{ch} \eta l \cos \nu l - \frac{1}{2}\left(\frac{\eta}{\nu} - \frac{\nu}{\eta}\right) \operatorname{sh} \eta l \sin \nu l = 1. \qquad (121)$$

where

$$\eta = \varkappa^2 r_u \sqrt{-\frac{\omega}{2} + \sqrt{\frac{\omega^2}{4} + \frac{1}{\varkappa^4 r_u^4}}}; \qquad (122)$$

$$\nu = \varkappa^2 r_u \sqrt{\frac{\omega}{2} + \sqrt{\frac{\omega^2}{4} + \frac{1}{\varkappa^4 r_u^4}}}. \qquad (123)$$

Through equation (121) is found value $\varkappa$, and then they calculate

$$a=\varkappa l. \tag{124}$$

After substituting formula (124) into expression (72), is found the value of Young's modulus. Obtained in this way of the value of Young's modulus, they are shown on Fig. 39 (curve 2). Use of more complex differential equation gives close results (Fig. 39, curve 3).

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Chapter IV

ELASTICITY CHARACTERISTICS OF REFRACTORY METALS AND OF ALLOYS AT HIGH TEMPERATURES.

From known at present metals only a few have high melting points. This is a number of the transition metals, which are characterized by the high strength of interatomic communication/connections (Fig. 40). Name itself "high-melting" thus far is not yet completely determined. Frequently the high-melting ones are considered all metals the melting point of which is higher than 1805°K (melting point of iron) [52], and sometimes only those the melting point of which is higher than 2670°K: tungsten, rhenium, tantalum, osmium, molybdenum, niobium, iridium. From the enumerated metals most widely are utilized tungsten, molybdenum, niobium and tantalum (the so-called large tetrad), which because of their high melting point and sufficient abundance are promising ones during the creation of structural materials for a work about very high temperatures.

Table 4. Fundamental physical characteristics of refractory metals [52, 62, 70].

(1) Физическая характеристика	Nb	Ta	Mo	W
(2) Атомный номер	41	73	42	74
(3) Атомный вес	92,91	180,95	95,94	183,85
(4) Атомный объем	10,8	10,9	9,41	9,53
(5) Междуатомное расстояние, Å	2,859	2,859	3,1403	3,1583
(6) Атомный радиус, Å	1,45	1,46	1,40	1,41
(7) Тип кристаллической решетки	ОЦК	ОЦК	ОЦК	ОЦК
(8) Коэффициент упругой анизотропии	0,49	1,58	0,906	1,008
(9) Коэффициент термического расширения при 293° К, $\alpha \cdot 10^{-6}$	7,1	6,6	5,44	4,45
(10) Плотность, $кг/м^3 \cdot 10^{-3}$	8,57	16,6	10,20	19,23
(11) Температура плавления, °К	2770	3270	2880	3650

Key: (1). Physical characteristic. (2). Atcmic number. (3). Atomic weight. (4). Atomic volume. (5). Interatomic distance, Å. (6). Atomic radius, Å. (7). Type of crystal lattice. (8). Cceefficient of elastic anisotropy. (9). Thermal-expansion coefficient cf 293°K, $\alpha \cdot 10^{-6}$. (10). Density, $kg/m^3 \cdot 10^{-3}$. (11). Melting pcint, °K.

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Table 4 gives some fundamental physical characteristics of these metals. The observed similarity in the properties of tungsten and molybdenum, and also of nicbium and tantalum is determined by the uniformity of the structure of their electron shells, since the metals indicated are the cell/elements of the identical groups of the periodic system of D. I. Mendeleyev (W, Mo - VI group, Nb, Ta - V - a

group). Elasticity characteristics are not exception/elimination from this general law; therefore below they will be examined respectively in each pair of these metals.

It is necessary to note that the physicomechanical properties of real metal depend to a considerable extent on the method of its obtaining, purity/finish and state (work-hardened, annealed, recrystallized). Since the refractory metals under industrial conditions are obtained by the methods of powder metallurgy (although recently is already familiar their melting in arc and electron-beam furnaces), the values of elasticity characteristics of the metal of different production can differ. Comparable can be only such values E, G, μ, which are measured one and the same dynamic method in the specimen/samples of one compositions and states.

Together with the application/use of refractory metals, which have industrial purity/finish and finding in polycrystalline state, expands the field of application of parts, manufactured from the single crystals of these metals. It is establish/installed, that the single crystals of the tungsten, molybdenum and other refractory metals in the state of high purity/finish do not lose plasticity up to the temperature of liquid helium [52]. The modern crystal taking into account elastic anisotropy has the only system of elastic constants. For cubic crystals, for example, a number of elastic

constants. For cubic crystals, for example, a number of elastic constants is equal to three. The value of Young's modulus and displacement in single crystal will depend on the direction of their measurement. The values of Young's modulus and displacement can be calculated according to the values of elastic constants, and vice versa [36].

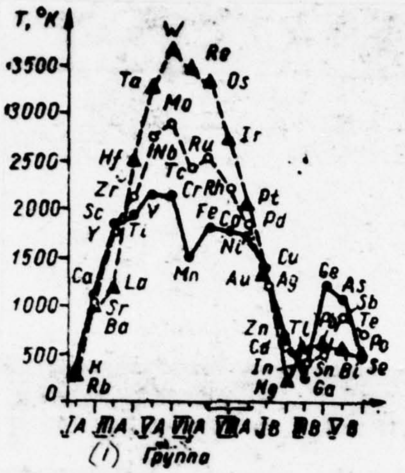

Fig. 40. Change in the melting point of transition metals in dependence on their position in periodic system.

Key: (1). Group.

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All this must be taken into consideration, if the determination of the module/moduli of the first and second kind is produced in the specimen/samples, manufactured from single-crystal blanks.

At present considerable effort/forces are directed toward the development of different alloys of refractory metals. In alloys it is possible to obtain such successful combination of different properties, which possesses not one pure metal. However, still it is

not possible to previously calculate the composition of such alloy which has the required properties. Precise information about the properties of alloy, including about elastic properties, can be obtained only as a result of experimental investigations.

1. Tungsten, molybdenum and their alloys.

Tungsten. Conducted in recent years numerous investigations showed that of the refractory metals of "large tetrad" the tungsten possesses the best strength properties at high temperatures. Even at 2300°K short-term strength of tungsten reaches about 100 N/mm^2 [46]. At room temperature both cast and sintered, the tungsten is brittle. The high temperature of transfer/transition from brittle state into plastic, that reaches in tungsten of 570-770°K, creates large difficulties during the manufacture from it of different parts. To shortcomings in the tungsten, one should relate also its high oxidizability during heating in air, and also high density.

Depending on the method of obtaining, the specific gravity/weight of this metal varies within the limits with 165-188 kN/m^3 [57]. On Young's modulus of tungsten at the normal temperature in the literature, most frequently are given V. Koster's data [79] (E=407 GN/m^2) which were obtained by measuring the resonance frequency of transverse vibrations of specimen/samples. M. G. Lozinskiy [33]

will determine by a similar method Young's modulus of cermet tungsten ($E=386\ GN/m^2$). By other researchers' majority who carry out measurements in the specimen/samples of the cermet tungsten of different porosity, were obtained the values of the module/modulus of the normal elasticity $E=344-412\ GN/m^2$ [34, 62, 63, 73, 82].

We have carried out the measurements of Young's modulus of cermet tungsten of the mark/brand of VRN of the production of Moscow electric bulb plant (specific gravity/weight 181-184 kN/m^3) [45]. Specimen/samples are made from the rods with a diameter of 8 mm by machining external surface on grinding machine with the subsequent facing. The results of measurements will show that the elastic modulus of this metal at the room temperature $E=390\ GN/m^2$. Extrapolation of the values of Young's modulus for poriferous tungsten for zero porosity on the data of work [34] gives $E \approx 407\ GN/m^2$.

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The value of Young's modulus have determined we and V. A. Dreshpak in specimen/samples made of the cast tungsten, smelted in electric-arc furnace, and also the tungsten, which passed cathode-ray remelting [14, 22]. Ingots were subjected to plastic deformation at the high temperatures and to annealing in vacuum. Specimen/samples

are made from the rod with a diameter of 10 mm by machining on lathe with the following by grinding emery paper. Young's modulus of the specimen/samples made of tungsten, manufactured from the ingots of electric arc remelting, will render/show equal to 407 GN/m², and obtained from the ingots of cathode-ray remelting - 404 GN/m².

Much less than information is about the modulus of shear of tungsten. On measurement data of V. Koster [79], in tungsten at the normal temperature G=148 GN/m². In later works are given the values, close to that indicated [62]. The cermet tungsten of the mark/brand of VRN on data of our measurements has shear modulus at the normal temperature G=145 GN/m², while in the cast tungsten G=160 GN/m².

The Poisson ratio of tungsten, according to V. Koster's data [79], is equal to 0.30. This value is given for tungsten and in handbooks [62, 63], although the value of the Poisson ratio of tungsten, calculated in the values of his moduli of elasticity, by specific V. Koster, is equal to 0.37. On measurement data by A. B. Lyashchenko [34] the Poisson ratio of tungsten μ=0.31.

The calculated by us in the values of the moduli of elasticity value of Poisson ratio of cermet tungsten of the mark/brand of VRN is equal to 0.34, and the cast tungsten - 0.26. In pulled tungsten wire the value of the moduli of elasticity depends to a considerable

extent on diameter and temperature of annealing [1, 56]. The value of Young's modulus varies within the limits of 147-372 GN/m², while that of shear modulus - within the limits of 137-177 GN/m². P. Wright [1] investigates in detail the elastic properties of single-crystal tungsten wire with a diameter of 0.9 mm. He will establish that the crystals of tungsten are almost isotropic. Differences in the elastic properties of crystals are very insignificant (less than 0.2o/o). Regarding P. Wright, Young's modulus of single-crystal tungsten wire is equal to 386 GN/m², and shear modulus - 150 GN/m². B. Chalmers [68] will give the value of the modulus of normal elasticity of the single crystals of tungsten in directions [111] and [100], equal to 368 GN/m², while that of shear modulus - 150 GN/m² will make the conclusion that in this metal was inherent the isotropic elasticity. V. A. Dreshpak will determine elasticity characteristics of the single crystal of tungsten in the test sample with a diameter of 7 mm and with a length of 73 mm at room temperature. The preparation for specimen/sample produces as follows.

The grown single crystal mechanically is worked on plain grinding machine and is polished by diamond disk.

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Such obtained thus test sample was subjected to chemical etching in

the 3o/o opening of percxide of hydrogen with the small addition of ammonia for relieving the surface work hardening. As a result of etching over an entire surface of specimen/sample, was removed the layer with a thickness of 0.1 mm. The crystallographic orientation of the longitudinal axis of specimen/sample is determined by X-ray method. X-ray photographs are remove/taken from fixed specimen/sample, in this case, is applied the emission/radiation with continuous spectrum according to the Laue X-ray method during setting up URS-60 with molybdenum tube. In the investigated specimen/sample the longitudinal axis composes 16° with direction [100]. Young's modulus of this specimen/sample will prove to be equal to 390 GN/m², while shear modulus - 158 GN/m². The designed by the values of moduli of elasticity Poisson ratio was equal to 0.24.

Table 5. Elasticity characteristics of tungsten at different temperatures.

(1) Темпера-тура, °K	(2) Вольфрам металлокерамический			(3) Вольфрам литой (электронно-лучевого переплава)		
	(4) E, Гн/м²	(4) G, Гн/м²	μ	(4) E, Гн/м²	(4) G, Гн/м²	μ
273	390	145	0,34	404	160	0,264
370	385	143	0,34	400	159	0,260
470	380	141	0,34	396	157	0,261
570	376	139	0,35	393	155	0,269
670	372	137	0,35	389	154	0,265
770	368	135	0,35	385	152	0,269
870	363	133	0,35	381	150	0,270
970	349	132	0,35	377	148	0,274
1070	354	131	0,34	374	146	0,280
1170	350	129	0,35	370	145	0,280
1270	345	127	0,35	367	143	0,280
1370	341	125	0,35	363	141	0,285
1470	336	123	0,36	360	139	0,290
1570	331	122	0,36	355	137	0,293
1670	325	120	0,36	350	135	0,293
1770	314	117	0,34	344	133	0,290
1870	306	—	—	335	130	0,285
1970	295	—	—	328	127	0,288
2070	284	—	—	321	124	0,291
2170	274	—	—	314	122	0,291
2270	265	—	—	306	119	0,290
2370	255	—	—	298	116	0,290
2470	245	—	—	290	112	0,297
2570	—	—	—	283	110	0,290
2670	—	—	—	275	107	0,290
2770	—	—	—	268	104	0,290
2870	—	—	—	260	101	0,292
	—	—	—	153	—	—

Key: (1). Temperature, °K. (2). Tungsten cermet. (3). Tungsten cast (cathode-ray remelting). (4). GN/m².

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Young's modulus in tungsten at elevated temperatures was measured by resonance method by V. Koster at temperature to 1070°K

[79], by M. G. Lozinskiy at temperature to 1470°K [33], and recently by G. Braun and F. Armstrong [73] at temperature to 2670°K. In a pulse manner of the value of Young's modulus of tungsten were determined by B. Bernstein at temperature to 1470°K [72], and R. Lowry and A. Gones - to 2070°K [82]. By us together with V. A. Dreshpak also were measured the values of Young's modulus of cermet tungsten of the mark/brand of VRN [45] and of cast tungsten [14, 22] at high temperatures. The obtained temperature dependences of Young's modulus and their value through every 100 deg during heating are given in Table 5. In essence is observed a good agreement of values of Young's modulus of tungsten, our obtained by different researchers, and data on all temperature range (Fig. 41). Exception/elimination represent only the results of work [73], according to which even for normal temperature the value of Young's modulus of tungsten comprises more than 500 GN/m².

From those obtained hire data it follows that the difference between the values of Young's modulus of cast and cermet tungsten, that occurs at normal temperature, is maintained at high temperatures. The general character of the temperature dependence of Young's modulus of tungsten is not connected with the method of obtaining the metal. With an increase in the temperature of Young's modulus in tungsten, smoothly is depressed to 1770-1870°K (that it comprises $0{,}5T_{m}$), and then it falls more intensely. At high

temperatures the tungsten retains the high value of Young's modulus. So, at 2470°K Young's modulus of cermet tungsten of the mark/brand of VRN is equal to 245 GN/m², i.e., it is more than in steel at room temperature.

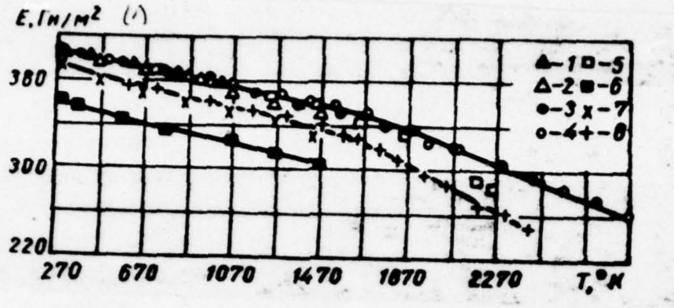

Fig. 41. The dependence of Young's modulus of tungsten on temperature according to different authors: 1 - [79]; 2 - [72]; 3 - [82]; cast tungsten; 4 - cathode-ray remelting [14]; 5 - electric arc remelting [22]; cermet tungsten; 6 - [34]; 7 - [33]; 8 - [45].

Key: (1). GN/m².

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The modulus of shear of tungsten with an increase in the temperature changes on the same laws, as Young's modulus. This one can see well from Fig. 42, where are given the temperature dependences of the modulus of shear of tungsten, obtained by different researchers, and the results of our own measurements. The values of the modulus of shear of the cermet and cast tungsten at different temperatures are given in Table 5.

The calculation of the Poisson ratio of tungsten conducted in

the values of Young's modulus and displacement shows that it barely changes with an increase in the temperature (Table 5).

Measurements described above of the moduli of elasticity of tungsten at high temperatures were carried out in polycrystalline specimen/samples. V. A. Dreshpak will determine the values of the moduli of elasticity at high temperatures in specimen/sample made of single-crystal tungsten. Measurements they conduct in the same specimen/sample, in which already were measured these module/moduli at normal temperature. The obtained values of Young's modulus and displacement of a single crystal of tungsten are represented in Fig. 43. After comparing the temperature dependences of the moduli of elasticity of single crystal and polycrystal of tungsten (Fig. 41-43), it is possible to draw the conclusion that they differ somewhat from each other. In single crystal the moduli of elasticity are decreased more evenly with heating on all temperature range and the intensity of an incidence/drop in them is less than in polycrystal.

From that examined it is possible to draw the conclusion that in tungsten at high temperatures the moduli of elasticity are retained higher than in any refractory metal which at normal temperature has moduli of elasticity higher than in tungsten (osmium, iridium, rhenium and ruthenium). With an increase in the temperature, an

incidence/drop in the mcduli of elasticity cf these metals occurs faster than in tungsten [79].

For describing the temperature dependences of the module/moduli of the elasticity both tungsten and other pure metals, by researchers' series were proposed different empirical expressions.

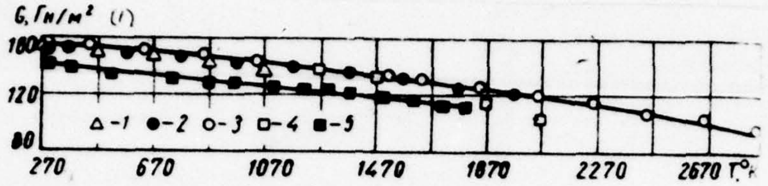

Fig. 42. The dependence of the modulus of shear of tungsten on temperature according to different authors: 1 - [72]; 2 - [82]; 3 - cast tungsten (cathode-ray remelting) [14]; 4 - [38]; cermet tungsten; 5 - VRN [45].

Key: (1). GN/m².

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For example, Hayes as early as 1923 [56] gave the expressions which describe the temperature dependences of the moduli of elasticity of the first and second kind of the monocrystalline tungsten wires:

$$E_T = E_0\left(\frac{T_s - T}{T_s}\right)^{0,263}; \quad (125)$$

$$G_T = G_0\left(\frac{T - T}{T_s}\right)^{0,263}, \quad (126)$$

where T_s - melting point, °K; E_0 and G_0 - respectively the value of Young's modulus and displacement at temperature of 0°K; E_T and G_T - respectively the value of Young's modulus and displacement at temperature T°K.

Carried out by us calculations showed that cnly expression for shear modulus gives satisfactory agreement with experimental data at low temperatures. For the values of Young's modulus, a good agreement of calculation data with experimental ones is obtained according to the expression

$$E_T = E_0\left(\frac{T_s - T}{T_s}\right)^{0.4}. \qquad (127)$$

Molybdenum. Molybdenum - refractory metal which most frequently applies for manufacturing of parts, workers at high temperatures. Although it is inferior to tungsten according to the melting point and high-temperature strength, for those cases when operating temperatures do not exceed 1970 - 2300°K, predcminantly is utilized molybdenum.

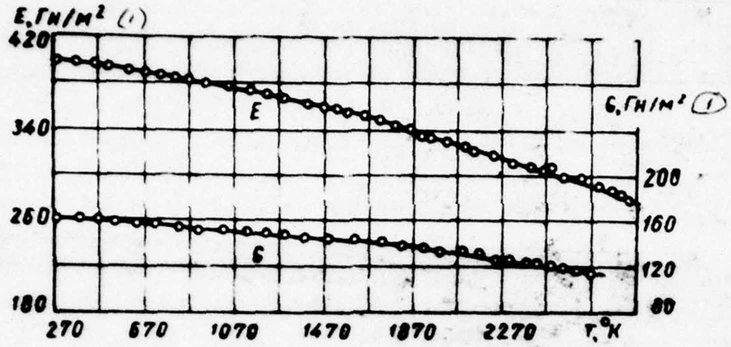

Fig. 43. Dependence of Young's modulus and displacement of the single crystal of tungsten on temperature.

Key: (1). GN/m².

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This is explained to the mainly fact that the molybdenum in comparison with tungsten possesses much smaller specific gravity/weight (100 kN/m³), but its machining does not represent difficulties, since the temperature of its transfer/transition into brittle state is close to room.

Molybdenum, as tungsten, possesses the high values of the moduli of elasticity. On measurement data of V. Koster [79], Young's modulus of molybdenum at room temperature is equal to 329 GN/m². The values of Young's modulus of molybdenum, obtained by other researchers, are

within the limits of 310-333 GN/m^2 [1, 33, 34, 63].

In work [63] for the molybdenum, obtained by the methods of powder metallurgy, is indicated Young's modulus, equal to 314 GN/m^2, and for the cast molybdenum - 317 GN/m^2. E. N. Marmer [38] on the basis of the analysis of the available data on Young's modulus of molybdenum at normal temperature recommends the utilizing of for calculations his value, the equal to 317 GN/m^2. We have carried out the measurements of Young's modulus of the molybdenum of the mark/brand of MRN, production of Moscow electric bulb plant (specific gravity/weight 98-100 kN/m^3 [20]. Specimen/samples for investigation are manufactured from the rods with a diameter of 8 mm via machining on lathe and annealing they do not undergo. The specific at normal temperature value of Young's modulus of these specimen/samples will prove to be equal to 322 GN/m^2. Young's modulus of cast molybdenum on the measurements, carried out by V. A. Dreshpak during setting up UP-6 [14], is also equal to 322 GN/m^2.

For the modulus of shear of molybdenum in the literature, are given the values from 120 to 124 GN/m^2 [38, 66]. In the molybdenum of the mark/brand of MRN, the shear modulus on the data of the our measurements is equal to 121 GN/m^2 [20], while in that cast, on measurement data of V. A. Dreshpak [14],-119 GN/m^2.

According to literature data, the Poisson ratio of molybdenum is equal to 0.31 [52] and C.324 [66]. The calculated by us value μ in the values of Young's mcdulus and displacement in the molybdenum of the mark/brand of MRN is equal to 0.32, but in that cast - 0.34.

The given higher than information about elasticity characteristics of molytdenum were obtained in specimen/samples in the form of rods. In the mclybdenum wires of value their somewhat different. For polycrystalline molybdenum wire with a diameter of 0.5-1.0 mm were obtained values of Young's modulus, equal to 250-292 GN/m² and 325 GN/m², while for single-crystal - 291 GN/m² [63]. Shear modulus in polycrystalline molybdenum wire with a diameter of 0.041 mm according to work [1] is equal to 135-150 GN/m², and in single-crystal wire by diameter 0.0365 mm - 179 GN/m².

On the data of K.C. Aleksandrov and T.V.Ryzhovaya [2], in the single crystal of molybdenum the modulus of normal elasticity in the direction [111] is equal to 294 GN/m², in the direction [110] - 313 GN/m² and in the direction [100] - 347 GN/m².

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By V. A. Dreshpak were determined the characteristics of elasticity of the single crystal of mclybdenum in the test sample with a

diameter of 8 mm and with a length of 90 mm. The manufacture of specimen/sample made of the grown single crystal of molybdenum and the definition of its orientation is produced just as specimen/sample made of the single crystal of tungsten. As chemical etchant was used aqua regia. In ready specimen/sample the longitudinal axis composes angle of 2-6° with direction [111]. Young's modulus of this specimen/sample at normal temperature will render/show 285 GN/m² of the modulus of displacement - 104 GN/m².

The measurements of Young's modulus of molybdenum at high temperatures by resonance method were carried out by V. Koster, who will determine its values at temperature of 1070°K [79]. This same method by M. G. Lozinskiy with colleagues during setting up IMASh-6 will measure Young's modulus of molybdenum at temperature to 1470°K [33].

B. A. Kalugin and I. G. Mikhaylov, utilizing a pulse method, will conduct measurements at temperature to 2970°K [19]. Their results, and also results of other researchers who conduct similar research, were represented in Fig. 44. Is observed the sufficiently good agreement of the results of all measurements, carried out by resonance method, including obtained by us and V. A. Dreshpak for the cermet molybdenum of the mark/brand of MRN [20] and cast [14]. The values of Young's modulus of cermet and cast molybdenum, and also modulus of shear and Poisson ratio, obtained by us at different

temperatures, are given in Table 6.

Figure 45 depicts the temperature dependence of the modulus of shear of molybdenum, constructed according to the data of different researchers and the results of our measurements.

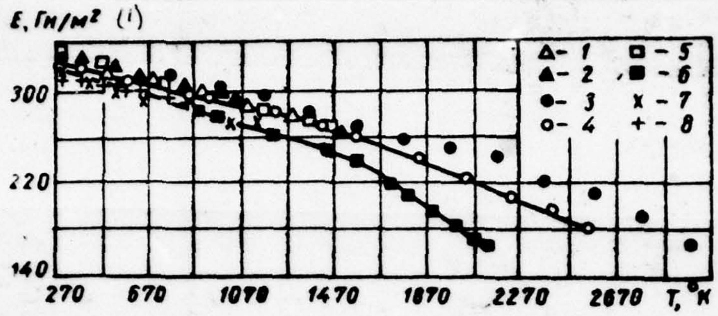

Fig. 44. The dependence of Young's modulus of molybdenum on the temperature: 1 - [79]; 2 - [33]; 3 - [19]; 4 - cast molybdenum (electric arc remelting) [14]; 5 - [34]; 7 - cast molybdenum [63]. Cermet molybdenum; 6 - [20]; 8 - [63].

Key: (1). GN/m².

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From the figure one can see that the character of the same its as the corresponding dependence of Young's modulus of this metal. The value of the modulus of shear of molybdenum, determined by pulse method at high temperatures [19], also differs from its values, obtained by resonance method.

Table 6 shows that the values of Poisson ratio barely change on all investigated temperature range. The determination of the values

of the moduli of elasticity at high temperatures in specimen/sample made of the single crystal of molybdenum is carried out by V. A. Dreshpak. Is utilized the same specimen/sample, in which were carried out the measurements of the values of elasticity characteristics at normal temperature. The findings on a change in Young's modulus and displacement of the single crystal of molybdenum with an increase in the temperature are represented in Fig. 46. The character of temperature dependences E and G has much in common with the character of the corresponding dependences in the single crystal of tungsten. The decrease of Young's modulus and modulus of shear of the single crystal of molybdenum passes approximately with identical intensity in all temperature interval.

Table 6. Elasticity characteristics of molybdenum at different temperatures.

(1) Темпера-тура, °K	(2) Молибден металлокерамический			(3) Молибден литой (электро-дуговой плавки)		
	(4) E, Гн/м²	(4) G, Гн/м²	μ	(4) E, Гн/м²	(4) G, Гн/м²	μ
293	322	121	0,32	322	119	0,335
370	315	120	0,31	318	118	0,350
470	311	118	0,32	314	116	0,355
570	306	116	0,32	310	114	0,360
670	300	114	0,31	306	112	0,366
770	294	112	0,31	301	111	0,360
870	289	110	0,31	297	110	0,352
970	282	107	0,32	292	108	0,355
1070	276	105	0,31	288	106	0,360
1170	270	103	0,31	284	105	0,354
1270	265	103	0,31	280	103	0,360
1370	258	98	0,32	275	101	0,364
1470	254	96	0,32	268	99	0,356
1570	248	94	0,31	263	96	0,366
1670	237	92	0,28	255	94	0,354
1770	222	—	—	247	91	0,354
1870	208	—	—	238	89	0,334
1970	193	—	—	231	87	0,341
2070	177	—	—	222	82	0,351
2170	163	—	—	216	79	0,358
2270	—	—	—	208	75	0,375
2370	—	—	—	200	—	—
2470	—	—	—	192	—	—
2570	—	—	—	184	—	—

Key: (1). Temperature, °K. (2). Molybdenum cermet. (3). Molybdenum cast (electric arc melting). (4). GN/m².

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The intensity of an incidence/drop in the moduli of elasticity in the single crystal of molybdenum is less than in polycrystal, moreover in molybdenum this difference in intensities is greater than in tungsten, what is, probably, by the consequence of its higher elastic

anisotropy (see Fig. 44 and 46).

For the temperature dependence of Young's modulus of polycrystalline molybdenum of Touni [44] it will propose the expression

$$E_T = E_0[1 - K(T - 273)], \tag{128}$$

where T - temperature according to the absolute scale; E_0 - value of Young's modulus with 0°K.

Value K is within the limits by 0.0001-0.0002. Equation (128) is satisfied well only on the first section of temperature dependence (to 1570°K). By analogy with tungsten for the temperature dependence of the module/moduli of the first and second kind of molybdenum, more greatly are suitable the expressions of the form

$$E_T = E_0\left(\frac{T_s - T}{T_s}\right)^{0.463}; \tag{129}$$

$$G_T = G_0\left(\frac{T_s - T}{T_s}\right)^{0.465}. \tag{130}$$

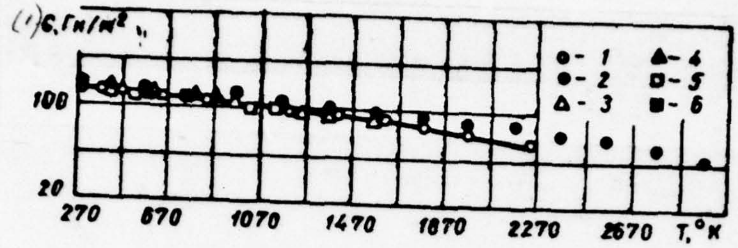

Fig. 45. The dependence cf the modulus of shear of molybdenum on temperature according tc different authors: 1 - cast molybdenum (cathode-ray remelting) [14]; 2 - [19]; 3 - [20]; 4 - [44]; 5 - cast molybdenum [63]; 6 - cermet molybdenum [63].

Key: (1). GN/m².

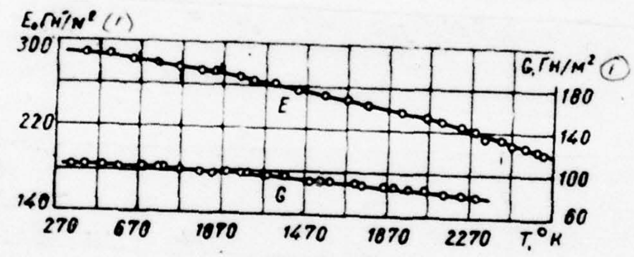

Fig. 46. Dependence of Ycung's modulus and displacement of single crystal of molybdenum or temperature.

Key: (1). GN/m².

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Alloys of tungsten and molybdenum. At present in the USSR and abroad intensely are conducted the developments of alloys on the basis of tungsten and molybdenum. There is information [52] that alloy W - 15o/o alloy Mc exceeds on strength the unalloyed tungsten with temperature to 2470°K. In practice will obtain thus far great distribution tungsten fusions with molybdenum, although it is already created and more compound alloys - triple and so forth. The strength properties of these alloys are investigated thus far still insufficiently in detail, but the experimental data on their elasticity characteristics there is no at normal and high temperatures generally.

By the author together with V. A. Dreshpak were determined elasticity characteristics of the tungsten-molybdenum alloys of different composition in the range of temperatures of 283-2970°K. These alloys were melted in electric-arc vacuum furnace. Then ingots were subjected to plastic deformation at high temperatures.

The moduli of elasticity of the alloy of one compositions are measured in two-three specimen/samples. The average values of elasticity characteristics of all investigated tungsten-molybdenum alloys at normal temperature are given in Table 7. From this table it follows that the value of Young's modulus and displacement of each alloy is proportional to the content in it of tungsten. The linear dependence of the moduli of elasticity on the content of components in tungsten-molybdenum alloys (Fig. 47) is explained those, that the tungsten and molybdenum possess the identical type of crystal lattice whose parameters differ altogether only to 0.57c/o, and is formed one with another the continuous number of solid solutions. At high temperatures the moduli of elasticity of tungsten-molybdenum alloys are determined on installations UP-5 and UP-6. The obtained temperature dependences of Young's modulus in the alloys of different composition are represented on Fig. 48, and modulus of shear - on Fig. 49.

Table 7. Elasticity characteristics of tungsten-molybdenum alloys.

(1) Состав сплавов, ат. %	(2) E, Гн/м²	(2) G, Гн/м²	μ
W+52 Mo	401	157	0,27
W+20,8 Mo	389	152	0,27
W+39,5 Mo	372	147	0,27
W+50,1 Mo	362	142	0,28
W+60,1 Mo	350	139	0,27
W+79,5 Mo	389.	123	0,32

Key: (1). Composition of alloys, at.%. (2). H/m^2.

With an increase in the temperature, the moduli of elasticity in all alloys are decreased first slowly (approximately to 0.5 $T_{пл}$), and then are faster. On temperature dependences of shear modulus, this change in the rate of an incidence/drop in the value of module/modulus is noticeably less clearly than to temperature dependences of Young's modulus.

By V. A. Dreshpak are also determined the characteristics of the elasticity of tungsten fusion with rhenium in the range of temperatures of 2903-2970°K. At room temperature this alloy, which contains 27 weights o/o rhenium, has Young's modulus 404 H/m^2, modulus of shear 154 H/m^2 and these calculated in two values Poisson ratio - 0.30.

The values of the moduli of elasticity with high temperatures of alloy W+27o/o Re are represented on Fig. 50. From this figure it is evident that a change in Young's modulus and shear modulus with an increase in the temperature according to the character of curves recalls a change in them in pure tungsten, but the intensity of this change in this alloy is more. This is connected, probably, with the effect of the rhenium, whose moduli of elasticity with heating are decreased much faster than in tungsten.

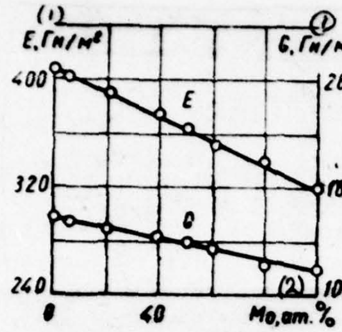

Fig. 47. Dependence of the moduli of elasticity on the content of components in tungsten-molybdenum alloys.

Key: (1). H/m². (2). Mo, at. o/o.

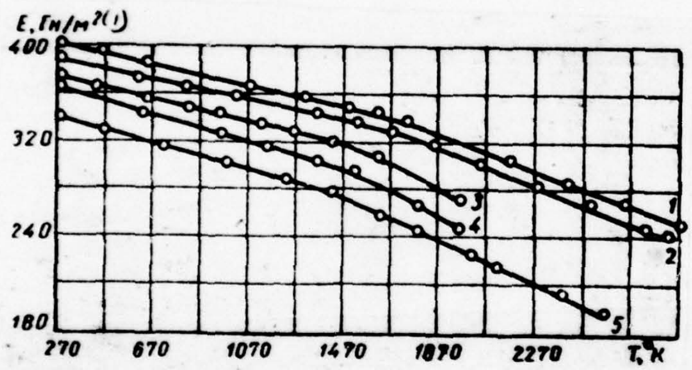

Fig. 48. The dependence of Young's modulus on temperature in tungsten-molybdenum alloys: 1 - W+5.2o/o Mo; 2 - W+20.8o/o Mo; 3 - W+39.5o/o Mo; 4 - W+50.1o/o Mo; 5 - W+79.5o/o Mo.

Key: (1). H/m².

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Examining the temperature dependences of Young's modulus and modulus of shear of tungsten, molybdenum and their alloys which were obtained by dynamic resonance method it is possible to note that their character was identical. They all take the form of descending curved with bend in region of temperatures 0.5 $T_{пл}$. The same law governing a change in the moduli of elasticity is observed also in the majority of other metals, if measurements are produced by dynamic resonance method [79].

The bends on of the curved, showing dependence moduli of elasticity on temperature, can express either change of the strength of interatomic connections in crystal lattice or course in the material of relaxation processes at this temperature and with loading. In the latter case during the measurement of the moduli of elasticity at the more high frequency of their value they will be obtained above, since the effect of relaxation processes will manifest itself to a lesser degree.

Exponential in this respect is the comparison of the temperature dependence of Young's modulus of the molybdenum, obtained by us resonance method (frequency on the order of 5 kHz), with the temperature dependence of Young's modulus of this metal [19], obtained by pulse method (frequency on the order of 5 MHz).

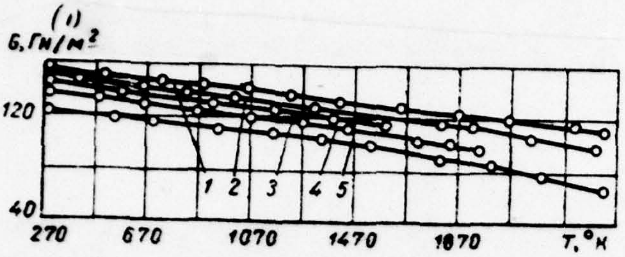

Fig. 49. The dependence of shear modulus on temperature in the tungsten-molybdenum alloys: 1 - W+5.2o/o Mo; 2 - W+20.8o/o Mo; 3 - W+39.5o/o Mo; 4 - W+50.1o/o Mo; 5 - W+79.5o/o Mo.

Key: (1). H/m².

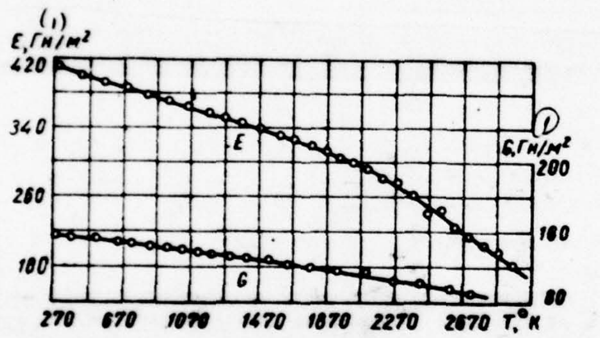

Fig. 50. Dependence of the moduli of elasticity on temperature in tungsten-rhenic alloy.

Key: (1). H/m².

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Both dependences are given in Fig. 51. From this figure it is evident that on the curved temperature dependence of Young's modulus, obtained at more high frequency, there is not discontinuity in temperature range 0.5 $T_{m.}$ although it has curvilinear character.

Thus, it can be assumed that the presence of the bends on curves of temperature dependences of the moduli of elasticity, obtained by resonance method at temperature 0.5 $T_{m.}$ in tungsten, molybdenum and their alloys is connected with the relaxation processes, which take place at this temperature, which in turn, are connected with the behavior of grain boundaries, since on the temperature dependences of the single crystals of tungsten and molybdenum such bends are not observed.

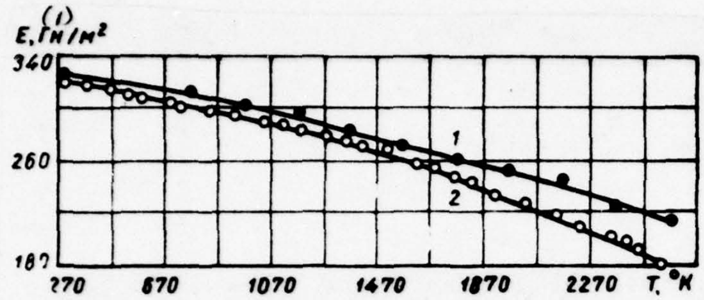

Fig. 51. The dependence of Young's modulus on temperature in the molybdenum: 1 - measurement by pulse method; 2 - measurement by resonance method.

Key: (1). H/m².

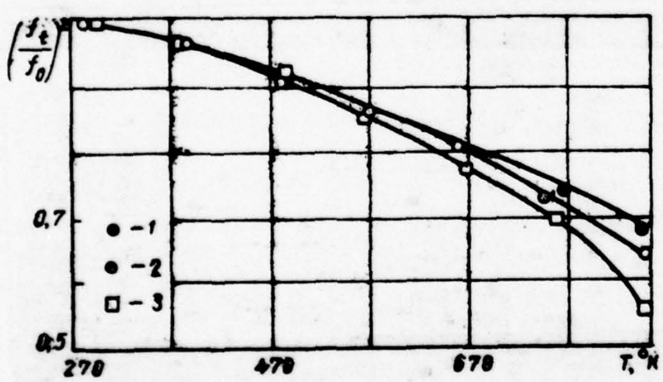

Fig. 52. The temperature dependence of Young's modulus of aluminum of different state: 1 - monocrystalline; 2 - polygonized; 3 - recrystallized.

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As confirmation of this can serve the results, obtained in work [7]. By the authors of this work were obtained the temperature dependences of Young's modulus of aluminum (99.99o/o Al) in different structural states. Investigations are carried out by resonance dynamic method in one and the same specimen/sample, having at first single-crystal structure, then the polygonized and finally polycrystalline coarse-grained. The polygonized structure was obtained by compressive strain (1o/o) along the axle/axis of single-crystal specimen/sample [110] with the subsequent annealing in vacuum with 870°K for 1 h. Coarse-grained polycrystalline structure was obtained via the annealing of the polygonized specimen/samples, additionally deformed by bend and again rectified. With an increase in the temperature in the single crystal of aluminum, is observed the steady decrease of Young's modulus (on Fig. 52 shown a change in proportional value f_t/f_0). To 300°K the character of change in the temperature dependence of Young's modulus is identical for all three structural states of aluminum.

At higher temperatures the decrease of Young's modulus occurs more intense than less modern is the material of specimen/sample. A similar character of curved temperature dependence of Young's modulus of aluminum, which is found in different structural state, in the

opinion of the authors of work [7], is connected with the increase of the role of inelastic phenomena on interfaces.

2. Niobium, alloys of niobium and tantalum.

Niobium. Among the refractory metals of "large tetrad" the niobium occupies special place. Although the melting point of its is lower than tungsten, tantalum and molybdenum, it possesses this combination of properties, that in many instances to it is loosened the preference before these metals. Especially valuable is its low capability for neutron capture. This property in combination with small thermal conductivity, good heat resistance and high life in water and in molten metals, such as potassium, sodium, lithium, being heat carriers in atomic reactors, will make niobium by irreplaceable material for nuclear power engineering.

The valuable qualities of niobium are its sufficiently high strength and plasticity at the normal and elevated temperatures, and also lower than in other refractory metals of "large tetrad", density. On heat resistance in the interval of temperatures 1300-1400°K, niobium is barely inferior to molybdenum in the cold-worked state and somewhat exceeds it in recrystallized state [17].

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Niobium is worked well at room temperature which is caused by its low temperature of transfer/transition from plastic into brittle state. During heating in air, the niobium is oxidized; however, it is considered that the problem of an increase in the oxidation resistance of niobium is considerably simpler than the problem of an increase in the oxidation resistances of molybdenum and tungsten, which form relatively low-melting and volatile oxides [5].

The enumerated above series of the properties of niobium shows that this metal is a promising structural material for many branches of technology. In connection with this to the study of niobium in recent years is given considerable attention. Are carried out numerous investigations of its physicomechanical properties. Some data given in works [17, 38, 41, 50], show that are observed the considerable disagreements as results of measurements of the physicomechanical properties of niobium, obtained by different by researchers, moreover disagreements concern not only the structurally dependent properties, such as the limit of strength, but also structurally independent variables, as for instance, the moduli of elasticity. Table 8 gives the encountered in the literature values of characteristics of elasticity of niobium at normal temperature, and also shows purity/finish, state of metal and applied method of

measuring the moduli of elasticity. For Young's modulus of niobium, were obtained the values from 85 tc 157 H/m². The highest value of the modulus of normal elasticity of niobium obtained in V. Koster's early work [78]. The purity of the metal which it uses, was not known. Data it considerably differ from those that were obtained during the subsequent years. As they correctly note I. M. Nedyukha and V. G. Chernyy [40], in later his work [79] V. Koster gives as tabulated data of M. Reycnods [86], considering them closer to real cnes.

The lowest value of Young's modulus for the niobium (85 H/m² will obtain K. Tottl [90] by the static method which, as is known, insufficiently was precise. The conducted by other researchers [41] cf measurement this same by method will become the already higher value of Young's modulus in niobium - 112 H/m². G. V. Zakharovoyp with colleagues [17] it was establish/installed, that quenching increases the value of the modulus of elasticity of niobium. In metal in deformed and recrystallized state, it is equal to 108 H/m², but in that harden/tempered - 122 H/m².

R. Begli [41] reveal/detected that the value of Young's modulus cf niobium depends on the medium in which are produced the measurements. In air obtained the value of Young's modulus of niobium, equal to 112 H/m², and in vacuum - 122 H/m². Any explanations of the reasons for this disagreement run n are brought.

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Table 8. Elasticity characteristics of niobium with normal temperature.

(1) Автор	(2) Год	(3) Чистота и состояние металла	(4) Метод измерения характеристик упругости	(5) E, Гн/м^2	(5) G, Гн/м^2	μ
(6) В. Костер [78]	1948	—	(7) Динамический резонансный	157	58,8	0,35
(8) М. Рейнольдс [86]	1953	(9) Отожжен в вакууме в течение 1 ч при $T=1320°K$	(10) Динамический импульсный	104	37,4	0,38
(11) П. Баррет, А. Сейболт [50]	1955	(12) То же	—	—	—	0,30
(13) К. Тоттл [90]	1957	(14) 99,95%, отожжен при 1570°K	(15) Статический	85	—	—
(16) Хазэльтон [77]	1958	—	(18) —	118	—	—
(17) Т. Хил [67]	1958	—	Динамический	104	—	—
(19) Д. Ливеси [81]	1959	—	.	108	—	—
(20) Р. Бегли [41]	1959	(14) 99,9%, отожжен при 1370°K	(21) Динамический (в вакууме)	122	—	—
(12) То же	1959	(12) То же	(22) Динамический (на воздухе)	112	—	—
(23) А. И. Дашковский, Е. А. Савицкий [12]	1960	—	(24) Динамический резонансный	—	40,7	—
(25) Г. В. Захарова и др. [17]	1961	(26) Д-формированный	(12) То же	108	—	—
(12) То же	1961	(27) Рекристаллизованный	. .	108	—	—
	1961	(28) Закаленный	. .	122	—	—
(29) Д. Лаверти, Е. Эванс [41]	1961	(14) 99,9%, отожжен при 1530°K	(15) Статический	112	—	—
(30) Ф. Остерман [85]	1962	—	(10) —	100	—	—
(31) И. М. Недюха, В. Г. Черный [40]	1965	99,4%	Динамический импульсный	104	37,2	0,398
(32) Н. Д. Тарасов, Р. А. Ульянов, (33) А. Д. Михайлов [58]	1965	—	(7) Динамический резонансный	108	—	—
(34) А. Б. Лященко [34]	1965	—	(12) То же	108	—	—
(35) Ф. Армстронг, Г. Браун [70]	1965	(37) —	. .	92	—	—
(36) В. А. Дрешнак [13]	1967	Отожжен в аргоне	. .	110	—	—

Key: (1). Author. (2). Year. (3). Purity/finish and state of metal. (4). Method of measuring elasticity characteristics. (5). H/m^2. (6). V. Koster. (7). Dynamic resonance. (8). M. Reinol'ds. (9). It is annealed in vacuum for 1 h with T=1320°K. (10). Dynamic pulse. (11). P. Barret, a. saybolt. (12). The same. (13). K. Tottl. (14). it is annealed when. (15). Static. (16). Hazelton. (17). T. Khil.

(18). Dynamic. (19). D. Livesi. (20). R. Begli. (21). Dynamic (in vacuum). (22). Dynamic (in air). (23). A. I. Dashkovskiy, Ye. A. Savitskiy. (24). Dynamic resonance. (25). G. V. Zakharov et al. (26). Deformed. (27). Recrystallized. (28). Harden/tempered. (29). D. Laverti, E. Evans. (30). F. Osterman. (31). I. M. Nedyukh, V. G. Chernyy. (32). N. D. Tarasov, R. A. Ulianov. (33). A. D. Mikhaylov. (34). A. B. Lyashchenko. (35). F. Armstrong, G. Braun. (36). A. Dreshpak. (37). It is annealed in argon.

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We have determined Young's modulus of compact and poriferous niobium, obtained by the methods of powder metallurgy (P=10o/o). In niobium there are impurity/admixtures: tantalum 0.25o/o, carbon 0.14o/o, silicon to 0.09o/o of iron 0.02o/o. Gas analysis is not conducted. Measurements are produced in specimen/samples in the nonannealed state.

On the data of our measurements, Young's modulus of compact niobium is equal to 103 H/m², and poriferous - 78.5 H/m². Niobium the passed electric arc remelting, has Young's modulus 102 H/m². Determined by V. A. Dreshpak [13] Young's modulus of the niobium, screwed tighter to cathode-ray remelting, will render/show equal to 110 H/m².

Thus, in niobium is observed considerable scatter in the values of the modulus of normal elasticity with room temperature. For the modulus of shear of this metal, there is a considerably smaller quantity of experimental data, than for Young's modulus.; however, even for it are indicated the values, which differ one from another. Thus, for instance, on the measurements of M. Reynolds [86], of i. of M. Nedyukhya and V. G. Chernyy [40] the modulus of shear of niobium is equal to 37.2 H/m², A. I. Dashkovskogo, Ye. A. Savitskiy [12] - 40.6 H/m², V. Koster [79] - 58 H/m², but on handbooks [62, 63] - 86.5 H/m². The determined by us the modulus of shear of compact niobium is equal to 38.2 H/m².

For the Poisson ratio of niobium in the literature, are encountered values 0.30 [50], 0.35 [79], 0.38 [34, 86], 0.398 [40]. It should be noted that in a series of works, for example [53], for niobium are indicated such values of Young's modulus, the modulus of shear and Poisson ratio, which do not satisfy the known relationship/ratio between these values for an isotropic material. A change in the modulus of normal elasticity of niobium during heating is investigated by T. Khil [67] at temperature to 770°K, Hazelton [77] - to 820°K, R. Begli [41] to 1070°K, by M. G. Lozinskiy and by G. V. Zakharova [17] and A. B. Lyashchenko [34] - to 1470°K, N. D.

Tarasov, R. A. Ulianov, Ya. D. Mikhaylov [58] to 1300°K, F. Armstronoym and G. Braun [70] to 2070°K, F. Osterman [85] to 2270°K. The curves of temperature dependences different authors' data are shown on Fig. 53. In this same figure is given obtained by us temperature dependences of Young's modulus of compact niobium and niobium of electric arc remelting at temperature to 1870°K, and also the obtained by V. A. Dreshpak temperature dependences of Young's modulus of the niobium, subjected to cathode-ray remelting at temperature to 2470°K. Table 9 gives their values through every 100 deg during heating. Although almost all obtained temperature dependences of Young's modulus differ from each other, it is possible to note which majorities on them shows a very small change of the module/modulus in the range of temperatures from room to 1470°K.

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In this temperature interval of the value of Young's modulus of niobium not only they are not decreased as in the majority of metals, but sometimes even somewhat they grow/rise. It is higher than temperature of 1470°K, according to data our investigations and results, obtained V. A. Dreshpak, values of Young's modulus begin slowly to be depressed. By us it was also establish/installed by measurements on compact niobium, that the shear modulus in this metal behaves similarly (see Fig. 53).

On the nature of this peculiar change in the modulus of elasticity of niobium with an increase in temperature, are at present several points. In work [17] is presented the hypothesis that similar behavior of niobium can be caused by the presence in it of interstitial impurities (mainly oxygen), which at high temperatures enter in solid solution and strengthen interatomic connections of niobium. This assumption is confirmed by saybolt's data [87] about the fact that the solubility of oxygen in niobium, which is found in solid state, considerably they increase with an increase in the temperature. On an increase in the values of the modulus of elasticity with an increase in the oxygen content in the analog of niobium - tantalum, testify the experimental data [76], given to Fig. 54.

P. Armstrong and G. Braun [70] explain the character of a change in Young's modulus of niobium during heating, being based on results of measuring the modulus of elasticity of the single crystals of this metal.

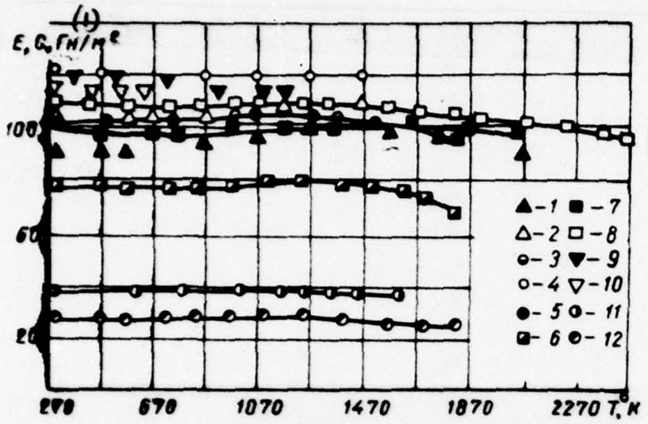

Fig. 53. Dependence of the moduli of elasticity of niobium on temperature by different authors' subject. Young's modulus; 1 - [70]; 2 - niobium deformed [17]; 3 - niobium recrystallized [17]; 4 - niobium harden/tempered [17]; 5 - niobium compact [20]; 6 - niobium poriferous [20]; 7 - niobium of electric arc remelting [13]; 8 - niobium of cathode-ray remelting [13]; 9 - testing in vacuum [41]; 10 - testing in air [41]. Shear modulus; 11 - niobium compact [20] 12 - niobium poriferous (our data).

Key: (1). H/m².

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By them establish/installed, that temperature dependence of Young's modulus in the specimen/samples whose longitudinal axis coincides

with direction [100], they differ in principle from temperature dependence of Young's modulus of specimen/samples with longitudinal axle/axis, which coincides with direction [110] and [111] (Fig. 55). In first specimen/samples with increase in temperature, Young's modulus is decreased, while for remaining increases. By Armstrong and Braun were carried out the experiments, which showed that in the polycrystalline recrystallized niobium with preferred texture in the direction [110] the character of the temperature dependence of Young's modulus was close to the same for single crystal [110] (Fig. 56). Braun they drew the conclusion that the character of the temperature dependence of Young's modulus of niobium is explained by the considerable elastic anisotropy of his crystals.

Table 9. Elasticity characteristics of niobium at different temperatures.

(1) Температура, °K	(2) Ниобий компактный			(3) Ниобий литой (электронно-лучевого переплава) E, Гн/м²
	(4) E, Гн/м²	(4) G, Гн/м²	μ	
293	103	38,2	0,35	110
370	103	38,2	0,35	109
470	103	39,2	0,31	109
570	103	39,2	0,31	109
670	103	39,2	0,31	108
770	103	39,2	0,31	108
870	104	39,2	0,33	108
970	104	39,2	0,33	109
1070	105	39,2	0,34	109
1170	105	39,2	0,34	110
1270	105	39,2	0,34	108
1370	104	39,2	0,33	108
1470	103	39,2	0,31	107
1570	102	38,2	0,33	107
1670	100	38,2	0,31	107
1770	98	—	—	106
1870	96	—	—	105
1970	—	—	—	104
2070	—	—	—	102
2170	—	—	—	101
2270	—	—	—	99
2370	—	—	—	98
2470	—	—	—	96

Key: (1). Temperature, °K. (2). Niobium compact. (3). Niobium cast (electron-beam remelting) E, H/m². (4). H/m².

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In work [34] is assumed that the reason for unusual behavior of Young's modulus of the riobium with of heating is the special structure of the electron shells of this metal. Since the available in the literature data on the values of the modulus of elasticity of

polycrystalline niobium at normal and high temperatures were obtained by larger part in specimen/samples with different structure and uncontrolled compositior, us will be undertaken the attempt to estimate the effect of structure and interstitial impurities (oxygen, nitrogen, carbon) on the value of the module/moduli of elasticity of niobium over a wide range of temperatures.

As initial material in all our investigaticns is utilized the niobium of double electron-beam melting. For explaining the degree of the effect of structure on modulus of elasticity, were made the specimen/samples made of forged rods (degree cf deformation 95o/o) and made of the rolled in one direction band (degree of deformation 65o/o.

Microstructures these samples are given in Fig. 57-a-d, and results of their tests - in Fig. 58. To temperature of 870°K, Young's modulus both of types of specimen/samples barely changes, with further increase in the temperature in forged metal, it is depressed, but in that rolled - it is raised. The temperature dependence of Young's modulus of rolled niobium is similar to the obtained F. Armstrong and G. Braun dependence of Young's modulus of single crystal with direction [110]. The reason for this similarity consists in the fact that, as shown in work [37], the structure of the rolled niobium approaches in orientation [110].

The part of the specimen/samples, manufactured from the rolled band, was subjected to recrystallization annealing (1 h at temperature of 1470°K).

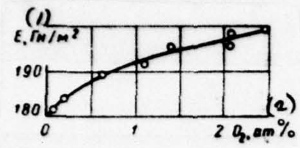

Fig. 54. Dependence of Young's modulus of tantalum on dissolved in it cxygen.

Key: (1). H/m². (2). at. c/o.

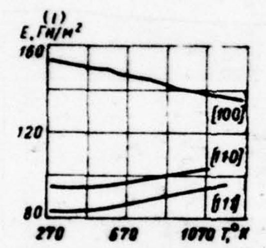

Fig. 5.

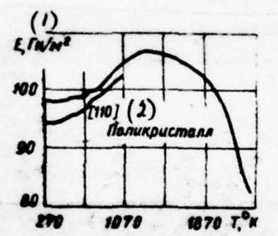

Fig. 6.

Fig. 55. The temperature dependences of Young's modulus of niobium, determined in single-crystal specimen/samples.

Key: (1). H/m².

Fig. 56. The temperature dependence of Young's modulus of polycrystalline niobium with the preferable arrangement of crystals in the direction [110] and single crystal of the same direction.

Key: (1). H/m². (2). Polycrystal.

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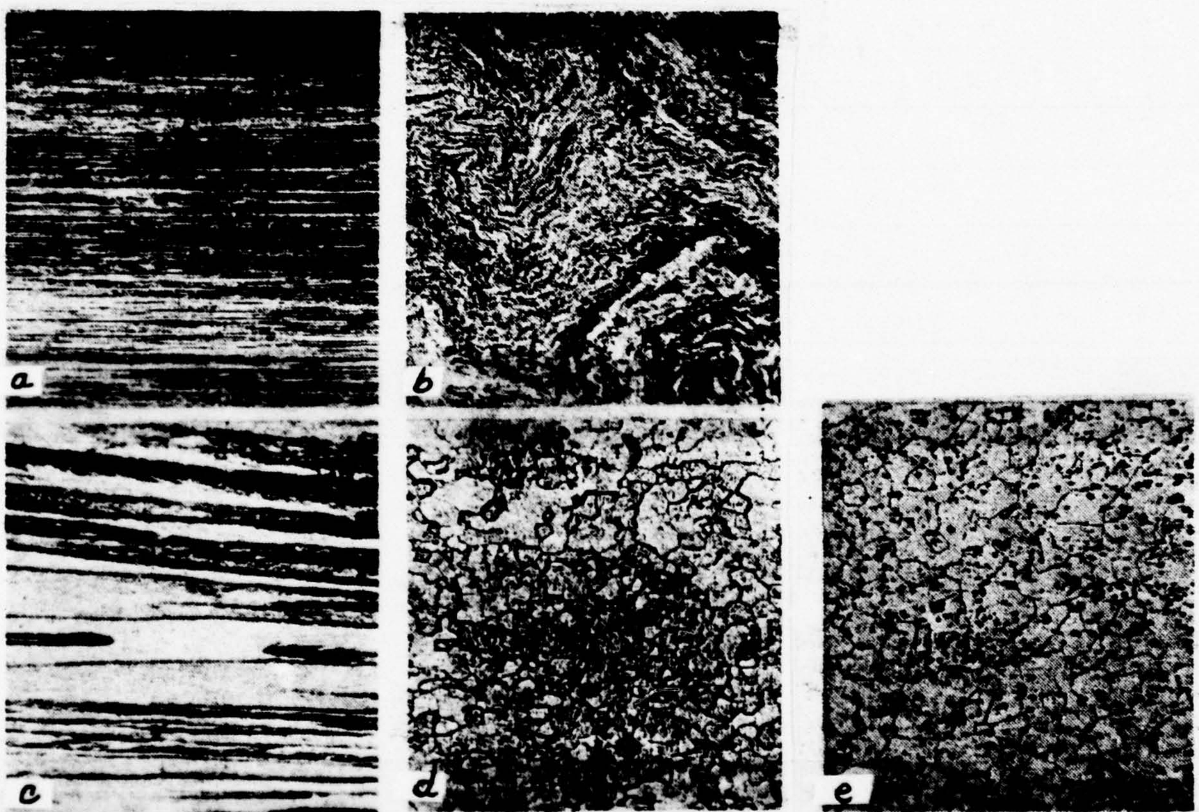

Fig. 57. Microstructures of the specimen/samples of the niobium: a and b - respectively along and across the direction of forging (x70): c and d - (x120); e - Nb+C.1o/o C (x 120).

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The temperature dependences of Young's modulus cf these specimen/samples (Fig. 58) turned cut to be differing little from the temperature dependences cf Young's modulus cf the specimen/samples, which did not pass this annealing. As will show data of microstructural analysis (Fig. 57d), this is connected with the fact that in such specimen/samples there exist the sections of deformation" banding",. already noted in work [71].

The study of the effect of the content of nitrogen and oxygen in niobium on its modulus cf elasticity is conducted in the specimen/samples, manufactured from rod. For obtaining different degree of saturation by oxygen these specimen/samples howl to annealed in vacuum $5 \cdot 10^{-4}$ mm Hg with temperature of 1420°K for 1 h (first batch), to annealing in the same vacuum at temperature of 2070°K for 1 h (second batch) and to oxidation in air for 3 h with temperature of 870°K with the water quenching with that following gone by in vacuum at temperature of 1420-1470°K for 3 h (third batch).

The temperature dependences of Young's modulus of the specimen/samples of all three batches are represented on Fig. 59. The comparison of curves shows that with an increase of oxygen and

nitrogen in niobium the decrease of Young's modulus with an increase in the temperature to 570-670°K becomes sharper.

The analysis of the effect of the carbon content in niobium on its modulus of elasticity for the first time carried out by V. Koster and V. Rausher in 1948 [80]:. They showed that an increase in the carbon content leads to an increase in the value of Young's modulus.

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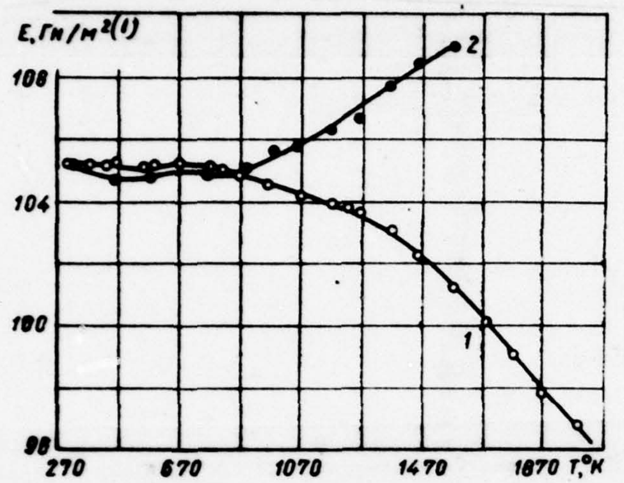

Fig. 58. Temperature dependences of Young's modulus of forged (1) and rolled (2) niobium.

Key: (1). H/m².

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In work [17] it is emphasized that these obtained researchers dependence one should consider as good-quality, since the authors of this work, probably, dealt with imperfect metal.

We have determined temperature dependences of the modulus of elasticity of niobium, containing 0.1o/o of carbon (Fig. 60). Carbon is inserted via burdening of the remelted molding/bars by carbide

titanium. With electron-beam remelting of titanium, it burns to high portions of percentage. Then ingot are hammered in air at the temperature of approximately 1270°K in the rod with a diameter of 12 mm with the degree of deformation 95o/o. The specimen/samples, manufactured from this rod, underwent tests in the cold-worked and recrystallized states (see Fig. 57e).

As it follows from findings, carbon raises the modulus of elasticity of niobium at temperature from room to 1270-1370°K. Further increase of the temperature of the niobium, saturated by carbon, causes a sharper incidence/drop in Young's modulus, than in pure metal.

Niobium fusions. Niobium is promising metal for creation on its basis of different heat-resistant alloys. As is indicated in work [50], to this they contribute its high melting point, the preservation/retention/maintaining of it by the alloys of high strength and plasticity with working temperatures higher than 1000°K, smaller specific gravity/weight and higher modulus of resistance with respect to weight, than in molybdenum, tantalum, tungsten, considerably the smaller activity of interaction with atmosphere, than in the latter, and the possibility of designing of the coatings, stable at high temperatures.

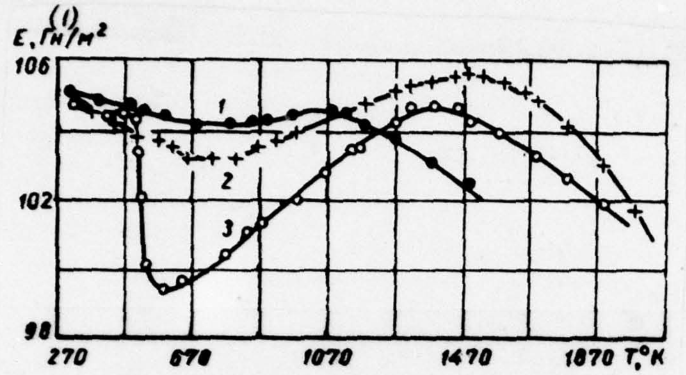

Fig. the temperature dependences of Young modulus of the niobium, which contains nitrogen and oxygen 1 - annealing with T=1420°K for 1 h; 2 - annealing with T=2070°K for 1 h; 3 - oxidation in air with T=870°K for 3 h, annealing in vacuum with T=1470°K for 3 h.

Key: (1). H/m².

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By the alloying of niobium can be raised the resistance of its creep at high temperatures and the resistance to oxidation, and are also improved plastic properties.

In work [58] are investigated the results of the effect of the alloying of niobium with small quantities of chromium, rhenium, tungsten, molybdenum, tantalum, iridium, palladium, zirconium and

titanium, i.e., the cell/elements, which form with niobium the unlimitedly solid solutions of pi all temperatures or having the sufficiently wide regions of solubility, on the character of interatomic interaction in solid solutions. On Fig. 61, are represented they are given in work [58] data about change values of the modulus of elasticity in dependence on the content of the alloying cell/elements. These dependences in essence are determined by the general laws, which occur during the formation of solid solutions. So, the alloying of niobium with chromium, rhenium, tungsten, molybdenum, tantalum, iridium, that have the higher modulus of elasticity, gives alloys, module/modulus of the elasticity they are which above than basic metal. The addition of zirconium and titanium whose module/moduli are close to the modulus of elasticity of niobium, it leads to an insignificant increase in the modulus of the elasticity of alloy (Zr) or its lowering.

Table 10, comprised on of works [13, 65], give corrected values of the modulus of elasticity of niobium fusions, which contain several alloying cell/elements. From this table it follows that in the dual and ternary alloys of niobium the dependence of the modulus of elasticity on form and quantity of alloying cell/elements is more complex, but the character of the effect of the alloying cell/elements on the modulus of elasticity is retained.

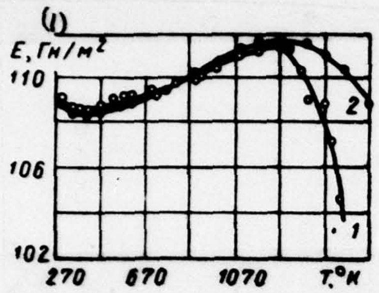

Fig. 60.

Fig. 61.

Fig. 60. The temperature dependences of Young's modulus of the niobium, which contains 0.1c/o of carbon: 1 - not annealed; 2 - annealed with T=1470°K for 1 h.

Key: (1). H/m².

Fig. 61. Dependence of the modulus of elasticity of niobium on the content of the alloying cell/elements.

Key: (1). H/m². (2). Alloying cell/element, weight o/o.

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By alloying the modulus of elasticity in niobium can be considerably raised. So, in alloy Nb+15o/o W+5c/o Mo+1o/o Zr Young's

modulus is equal to 172 N/m^2, which is almost 1.5 times higher than in the unalloyed metal. Consequently, niobium fusions can not have this shortcoming as the small value of the modulus of elasticity, which will considerably expand the region of their application/use.

Works [14, 58] also give the information about a change in the modulus of normal elasticity of niobium alloys during heating. According to data these works are constructed the temperature dependences of Young's modulus of some alloys of niobium (Fig. 62). As can be seen from figure, in all investigated alloys the temperature dependences of the modulus of elasticity have in essence the same character as in the unalloyed niobium. This, probably, is connected with the fact that the total quantity of those alloying cell/element in each alloy does not exceed 20o/c and they not we can significantly change the dependence of the modulus of elasticity on temperature.

Tantalum. From refractory metals which are sufficiently familiar and are applied in industry (W, Mo, Nb, Ta), tantalum thus far is utilized in small quantities. This is connected with its high cost/value, limited resource/lifetimes, and also high density. However, the high melting point of tantalum (3270°K), which higher than in molybdenum and niobium, and also its ability to remain plastic even at very low temperatures, makes tantalum with promising

structural material.

The mechanical properties of tantalum at high temperatures, especially its elasticity characteristics, thus far are still studied insufficiently fully.

Table 10. Moduli of normal elasticity of some alloys of niobium.

(1) Состав сплава	(2) Температура, °K	(3) Модуль Юнга, Гн/м²
Nb+15% W+5% Mo+1% Zr	293	172
То же	1365	123
Nb+15% W+5% Mo+5% Ti+1% Zr	293	165
То же	1365	113
Nb+10% Mo+10% Ti	1365	58,4
Nb+32,5% Ta+0,75% Zr	293	113
Nb+14% W+2% Mo	293	130
То же	1370	129
Nb+14% W+2% Mo+0,83% HfC	293	124
То же	1370	126

Key: (1). Composition of alloy. (2). Temperature, °K. (3). Young's modulus, Gn/m².

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There is a series of the works in which are given the results of the measurements of elasticity characteristics of tantalum at the normal temperatures: for Young's modulus, are obtained the values of 175-187 H/m² [34, 41, 79], for modulus of shear - 68.5 H/m² [37] and for Poisson ratio - 0.35 [53]. As concerns the information about the values of the characteristics of elasticity at high temperatures, then in literature are only data about values of Young's modulus at temperature of 1370°K [34, 71, 79].

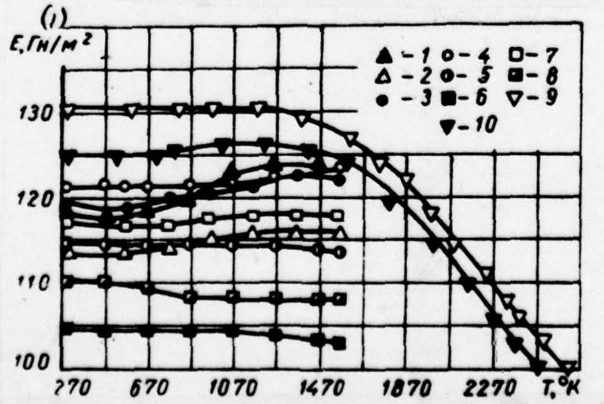

Fig. 62.

Fig. 62. The temperature dependences of Young's modulus of niobium fusion on data [58] 1 - Nb+10o/o Mc; 2 - Nb+10o/o Ta; 3 - Nb+10o/o W; 4 - Nb+5o/o Re; 5 - Nb+2.3o/o Cr; 6 - Nb+7.6o/o Ti; 7 - Nb+2.0o/o Ir; 8 - Nb+1.78o/o Zr; 9 - Nb+14o/o W+2o/o Mo; 10 - Nb+14o/o W+2o/o Mo+0.83c/o HfC.

Key: (1). H/m².

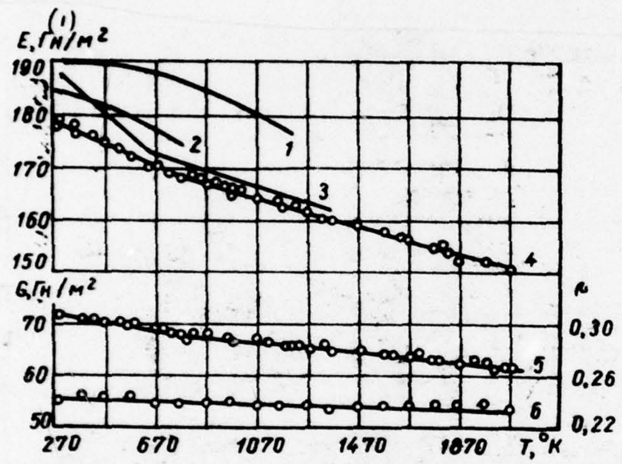

Fig. 63. The temperature dependences of characteristics of the elasticity of tantalum cn different authors' data. Young's modulus: 1 - [71]; 2 - [79]; 3 - [34]; 4 - our data; stear modulus; 5 - our data; Poisson ratio; 6 - our data.

Key: (1). H/m².

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We have determined elasticity characteristics of tantalum at higher temperatures.

From commercially pure tantalum of vacuum-arc melting, were made the specimen/samples with a diameter of 8 mm and with a length of 90 mm. Specific with normal temperature Young's modulus of tantalum will render/show equal to 175 H/m^2, modulus of shear 70.5 H/m^2, and Poisson ratio - 0.24. Measurements at high temperatures are produced in vacuum $5 \cdot 10^{-5}$-$5 \cdot 10^{-4}$ mm Hg. Obtained temperature dependences of elasticity characteristics of tantalum are given to Fig. 63, and their values through each 100 hail are given in Table 11. To Fig. 63, are plotted/applied also the temperature dependences of Young's modulus of this metal, obtained by other researchers.

Table 11. Elasticity characteristics of tantalum at different temperatures.

(1) Температура, °K	(2) E, Гн/м²	(2) G, Гн/м²	μ	(1) Температура, °K	(2) E, Гн/м²	(2) G, Гн/м²	μ
293	175	70,5	0,24	1270	158	63,5	0,24
370	173	69,5	0,24	1370	157	63,5	0,23
470	171	68,5	0,25	1470	156	62,7	0,24
570	169	68,5	0,24	1570	155	62,7	0,24
670	167	67,5	0,25	1670	153	61,8	0,24
770	165	66,5	0,24	1770	152	61,8	0,23
870	164	65,5	0,25	1870	150	60,8	0,23
970	162	65,5	0,23	1970	149	60,8	0,23
1070	161	64,5	0,24	2070	148	59,8	0,23
1170	160	64,5	0,24				

Key: (1). Temperature, K. (2). H/m².

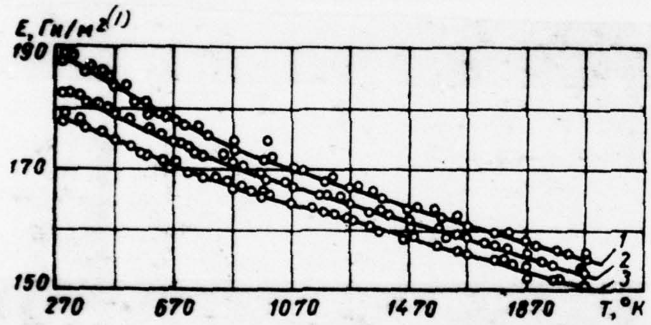

Fig. 64. The temperature dependences of Young's modulus of tantalum, saturated by oxygen: 1 - diffusion saturation by oxygen for 10 h; 2 - diffusion saturation by oxygen for 5 h; 3 - commercially pure tantalum.

Key: (1). H/m².

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The observed disagreement between the values of Young's modulus is connected, probably, with the fact that in each case is investigated tantalum, having different degree of contamination by impurity/admixtures, and also obtained by different methods. As early as 1955 by measurements at normal temperature [76] establish/installed the considerable effect of the impurity/admixture of oxygen on the modulus of elasticity of tantalum. So, with oxygen content 0.1 at. o/o Young's modulus of tantalum is equal to 176 H/m^2 and with oxygen content to 2.5 at. o/o values it is raised to 196 H/m^2.

We have determined the value of Young's modulus at high temperatures in the specimen/samples of tantalum, which were undergoing preliminarily diffusion saturation by oxygen. The preparation for specimen/samples is produced to 770°C, then one batch is age/held with this temperature for 5 h, and another batch - for 10 h. After this all specimen/samples pass annealing in vacuum with temperature of 1470°K for 1 h. In all led three cycles of this annealing. On Fig. 64, is shown temperature effect on Young's modulus of tantalum, which passed diffusion saturation by oxygen.

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Chapter V.

ELASTICITY CHARACTERISTICS OF METAL-LIKE AND NONMETALLIC MATERIALS AT HIGH TEMPERATURES.

1. Metal-like refractory compounds.

The materials, which possess high melting point, are the joints of transition metals with boron, carbon, nitrogen and silicon. They have high mechanical strength at high temperatures, considerable hardness and wear resistance, and also life against the action of molten metals, acids and alkalies. Like metals, metal-like refractory compounds have high electrical conductivity and thermal conductivity. Parts from these materials manufacture usually by the methods of powder metallurgy, which causes the presence in them of certain quantity of pores.

By their nature metal-like refractory compounds are brittle materials, especially at room and low temperatures. The conducted

investigations of the mechanical properties of refractory compounds [47] showed that in the latter is observed the essential difference between technical and theoretical strength and occurs the considerable dispersion/dissipation of test results. The strength properties of high-melting metal-like compounds also depend to a considerable extent on the form of loading and size/dimensions of test specimens. The strength of some refractory compounds at high temperatures is higher than with normal ones.

In spite of the noted shortcomings, refractory metal-like joints are found ever increasing use in high-temperature settings up because of special properties indicated above which do not possess the refractory metals and alloys.

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Elasticity characteristics of refractory compounds, especially at high temperatures, are studied still insufficiently. The values of Young's modulus for some joints (with zero porosity) at room temperature [54] are given below:

TiC	450	WC	695
ZrC	348	TiB_2	530
VC	428	ZrB_2	343
NbC	338	CrB_2	211
TaC	285	TiN	250
Mo_2C	533	$MoSi_2$	420
W_2C	420		

I. N. Frantsevich [64] will ncte that if the pure metals are very different in the strength cf interatomic bond (W and Ti), then their carbides are characterized virtually by the identical strength of interatomic bond. Transfer/transition frcm carbides to nitrides is accompanied by a very starp reduction/descert in the strength of this bond. By softening is accompanied also transfer/transition from one type of carbide to another (from WC to WC_2). The phenomena indicated affect the value of Young's modulus [54].

Us will be determined the temperature dependences of Young's modulus of several refractory compcunds of the production of the Institute of problems of the science of materials of the AS USSR. The specimen/samples of zirccnium diborid have different porosity (28-34o/o).

Table 12. Values of Young's modulus carbides at different temperatures.

(1) Темпера-тура, °K	SiC, П−11%	NbC, П−18%	TiC, П−19%	Mo_2C, П−26%
293	294	274	235	133
370	292	270	232	132
470	290	267	229	131
570	28[illegible]	260	225	130
670	286	258	221	129
770	284	255	218	128
870	282	250	214	128
970	280	247	210	127
1070	278	243	206	126
1170	276	239	202	126
1270	274	235	198	125
1370	270	232	194	125
1470	264	227	191	124
1570	260	223	186	122
1670	253	219	182	120
1770	246	215	176	117
1870	235	210	210	114
1970	224	203	165	—
2070	212	196	—	—

Key: (1). Temperature, °K.

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The results of measurements (Fig. 65) show that Young's modulus of this refractory compound with an increase in the temperature changes very insignificantly. Sc for ZrB_2 by porosity 32o/o at the room temperature E=150 H/m², and with T=1870°K E=123 H/m². The character of the temperature dependences of Young's modulus of zirconium boride of different porosity is almost identical.

The invariability of the character of temperature dependence during porosity change they are noted also by A. B. Lyashchenko, P. I. Melnichuk and I. N. Frantsevich [35] in carbide of titanium.

Figure 66 gives the results, obtained by these researchers, and given, obtained by us in the specimen/sample of carbide of titanium by porosity 19o/o. From given data it is possible to draw the conclusion that the porosity does not have considerable effect on the course of the temperature dependences of elasticity characteristics, but only is determined their absolute value.

By us are also carried out the measurements of Young's modulus at normal and high temperatures at carabides of niobium, molybdenum, rhenium and titanium. The obtained values of Young's modulus through every 100 deg during heating of these carbides are given in Table 12, and Fig. 67 shows the corresponding temperature dependences.

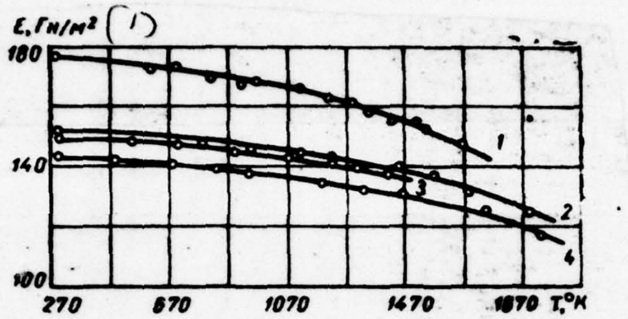

Fig. 65. The temperature dependences of Young's modulus of zirconium boride of different porosity (o/o): 1 - 28; 2 - 32; 3 - 38; 3 - 34.

Key: (1). E, H/m².

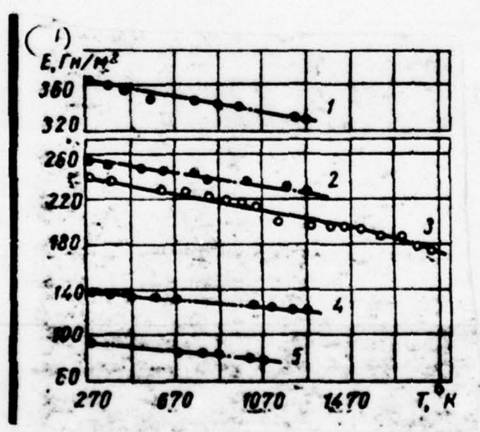

Fig. 66. Temperature dependences of Young's modulus of carbide of titanium of different porosity (o/c): On data [35] 1 - 7.8; 2 - 17; 4 - 32; 5 - 32; according to cur data by 3 - 19.

Key: (1). E, H/m².

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In all investigated carbides with an increase in the temperature, the moduli of elasticity are decreased very insignificantly. Especially vividly this is expressed in carbide of the molybdenum Young's modulus which at room temperature is equal to 133 H/m^2, and with $T=1770°K-117$ H/m^2.

Thus, in metal-like refractory compounds the moduli of elasticity are not only higher than in the appropriate metals (if we carry out comparison in nonporous state), but also much lesser they change during heating to high temperatures.

2. Nonmetallic refractory compounds.

Many properties of nonmetallic refractory compounds (carbides and nitrides of boron and silicon, boron compound and silicon between themselves and the like) are similar to the properties of metal-like refractory compounds, but in contrast the latter nonmetallic refractory compounds possess low electrical conductivity and thermal

conductivity. Their positive property is the ability to resist well thermal shocks.

There are assumptions [54] that with an increase in the temperature the moduli of elasticity of carbides and nitrides of boron and silicon change little, whereas in metal-like borides and carbides the temperature dependence of module/modulus is considerable.

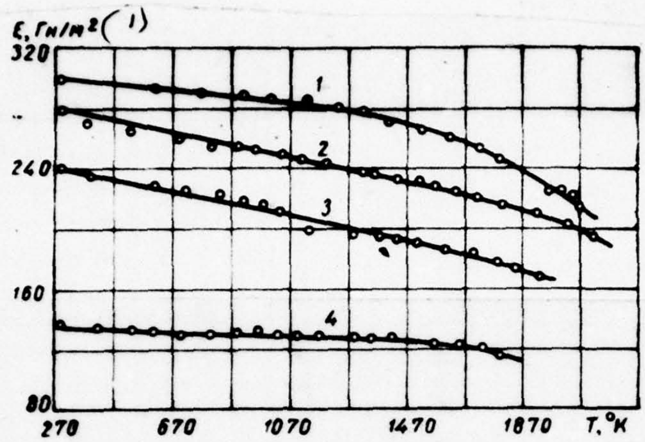

Fig. 67. The temperature dependences of the modulus of Young of carbides of silicon, niobium, titanium and molybdenum: 1 - SiC, P = 11o/o; 2 - NbC, P = 18o/o; 3 - TiC, P = 19o/o; 4 - Mo_2C, P = 26o/o.

Key: (1). E, H/m².

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This contradicts the information about the temperature dependences of Young's modulus of nitride of boron, obtained by K. Taylor [89], who observed considerably a reduction/descent in Young's modulus in this joint in the range of temperatures from normal to 1300°K (E=11.6 H/m² with 1300° K). Specimen/samples for these investigations were made of nitride of boron of α-phase and the measurements are produced in parallel to the direction of pressure forging.

We investigated a change in Young's modulus and displacement of the materials, which consist of the mixtures of nitride of boron and nitride of silicon, depending on the content of components, and also changes in values E and G with an increase in temperature [25]. For manufacturing the specimen/samples, are used the technical powders of nitride of boron and nitride of silicon with the average size of the particles of 15-20 μm. The chemical composition of powders is given in Table 13.

From the powders of nitride of boron and nitride of silicon, were prepared the mixtures with content by 20; 40; 60 and 80 mol.o/o of nitride of boron. Powders mix in ball mill for 24 h. After mixing to mixture, is added starch paste (200 g on 1 kg of mixture). Mixture dries on air, and then they rub through sieve with No 25 and press by the method of mouthpiece pressure forging. Blanks are sintered in resistance furnace with the graphite tube of heater in the current of nitrogen and the powder of nitride of boron with nitride of silicon (4:1) at temperature of 1870°K for 3 h. Ready specimen/samples take the form of the rods with a diameter of 10 mm and with a length of 90 mm they possess porosity 25-30o/o.

Young's modulus and displacement determine by measuring the

resonance frequencies of the transverse and torsional oscillations of specimen/samples. The value of Yourg's modulus and displacement of each mixture takes in terms of the averaged value of the results of measurements in four-five specimen/samples. The averaged values E and G were used for constructing the dependence of elasticity characteristics of material on the composition of components (Fig. 68).

Table 13. The chemical composition of nitrides of boron and silicon.

Порошок (1)	Содержание элементов, % (2)							
	$B_{общ}$	$Si_{общ}$	N	B_2O_3	$C_{общ}$	$C_{своб}$	Fe	Прочие (3)
Нитрид бора (4)	42,8	—	55,2	0,1	0,9	—	—	1,0
Нитрид кремния (5)	—	56,3	31,6	—	3,11	0,13	0,01	8,98

Key: (1). Powder. (2). Content of cell/elements, o/o. (3). Other. (4). Nitride of boron. (5). Nitride of silicon.

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The measurement of Young's modulus and displacement of specimen/samples at elevated temperatures is conducted with setting up UP-6. The obtained values of Young's modulus and displacement at different temperatures are shown on Fig. 69. First of all it should be noted that is observed very insignificant decrease E and G with an increase in the temperature in the materials, comprised of different mixtures of nitride of boron and nitride of silicon. In material with the increased content of nitride of boron, the decrease of the values of characteristics with temperature occurs less intensely, than in materials with the increased content of nitride of silicon. The

results, obtained by K. Taylor [89], will nct be confirmed, since by us is not observed such sharp reduction/descents in Young's modulus even in material with 80% ccntent of nitride of boron.

We carried out also the investigations of the effect of the addition of nitride of borcn on the elastic modulus of aluminosilicate refractcry. The selection of nitride of boron as additions to aluminosilicate refractory is explained by its high fire resistance and and by inertness in the absence of oxidation. Since the melting point (dissociation) of nitride of boron does not exceed 3000°K, articles made of nitride of boron do nct react with the molten glass and a number of metals.

As raw material for an aluminosilicate refractory, are utilized Chasov"yarsk clay of brands G-1 and TUO-51, the kaolin of the first type of Prosyanovkiy deposit. Fireclay manufactures from the mixture cf the refractory clay and kaolin with grain sizes: 1.5-0.5 mm - 55o/o; 0.5-0.1 mm - 29o/o; 0.1 mm are less - 16o/o. Nitride of boron has grain sizes less than 20 μm. Content of components in aluminosilicate refractcry following: clay - 24.7o/o; kaolin - 20.3o/o; fireclay - 55.0o/o.

From these materials were comprised the charges of this composition (weight o/o): aluminosilicate refractory - 90, nitride of

boron - 10; aluminosilicate refractory - 80, nitride of boron - 20; aluminosilicate refractory - 70, nitride of boron - 30; aluminosilicate refractory - 60, nitride of boron - 40; aluminosilicate refractory - 50, nitride, boron - 50; aluminosilicate refractory - 40, nitride of boron - 60. Specimen/samples press on hydraulic press at pressure 250 GN/cm^2. The humidity of charges during pressure forging composes 5-6o/o. The pressed specimen/samples after drying in cabinet drier are anneal/scorched in neutral gaseous medium to temperature of $1570^{\circ}K$ with holding at maximum temperature for one hour. Ready specimen/samples take the form of rods by size/dimension 150x15x15 mm.

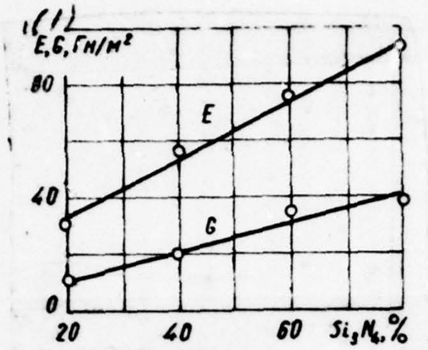

Fig. 68. Dependence of elasticity characteristics of materials from nitrides of boron and silicon on the content of nitride of silicon.

Key: (1). E, G, GN/m².

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At normal temperature Young's modulus determines with setting up UP-4. Great difficulties will arise as a result of the fact that the specimen/samples do not have clearly expressed resonance frequencies, and the considerable amplitudes of oscillations could not attain due to their high damping capacity. This, apparently, is connected with heavy graininess and heterogeneity of the material of specimen/samples.

For each composition the elastic modulus is determined in two-three specimen/samples. Figure 70 shows a change in Young's

modulus in aluminosilicate refractory with the addition in it of nitride of boron. The elastic modulus of initial aluminosilicate refractory will render/show equal in average/mean 34 GN/m^2. After the addition of nitride of boron, which possesses lower elasticity characteristics, than aluminosilicate refractory, we obtain material with lower elastic modulus. So, Young's modulus of material with 30o/o of nitride of boron is equal to 25.7 GN/m^2, and the elastic modulus of material with 60o/o of nitride of boron - 14.0 GN/m^2.

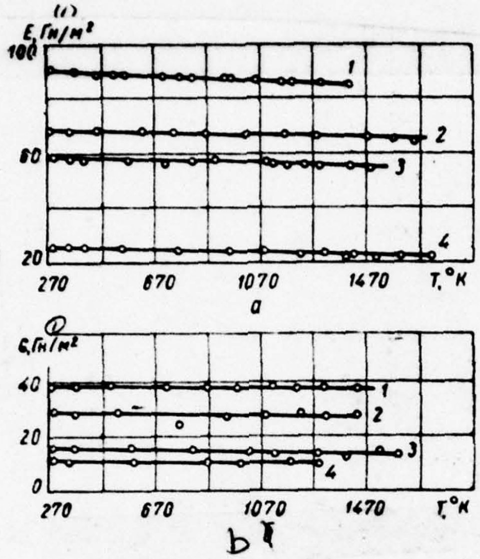

Fig. 69. The temperature dependences of Young's modulus (a) and of modulus of shear (b) of materials from nitrides of boron and silicon:

1 — 20% BN, 80% Si_3N_4; 2 — 40% BN, 60% Si_3N_4; 3 — 60% BN, 40% Si_3N_4; 4 — 80% BN, 20% Si_3N_4.

Key: (1). E, GN/m^2.

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Elastic modulus at the high temperatures was determined with setting up UP-6 in vacuum of $1 \cdot 10^{-4}$ mm Hg. In view of the fact that oscillation damping grow/rises with high temperatures, for an

increase in the amplitude of oscillations were made the specimen/samples of the lowered/reduced rigidity by size/dimensions 100x15x10 mm. Test the specimen/samples, not containing nitride of boron, or specimen/samples with 40o/o of nitride of boron. Measurements are conducted both during the heating and during cooling of specimen/samples. The obtained temperature dependences of elastic modulus are represented in Fig. 71. The character of the temperature dependence of Young's modulus of aluminosilicate refractory without the additions of nitride of boron is similar to that which is observed by other scientists [10]: stability to 870°K, and then insignificant increase. Young's modulus of aluminosilicate refractory with 40o/o of nitride of boron barely changes with an increase in the temperature.

3. Pyroceram and glass.

Vitreo-crystalline materials receive at present increasing propagation in technology because of their high strength, thermal and chemical stability. It is in detail elasticity characteristics of Pyrocerams yet not of emission/radiation, although it is establish/installed, that Young's modulus them is higher than in initial glasses, and it reaches 86-137 GN/m² [8].

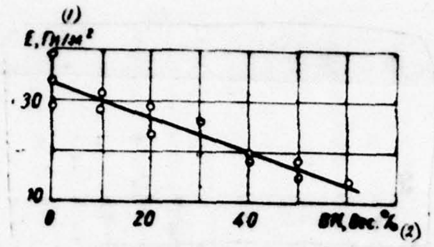

Fig. 70. Dependence of Young's modulus of aluminosilicate refractory on the content in it of nitride of boron.

Key: (1). E, GN/m². (2). weight %.

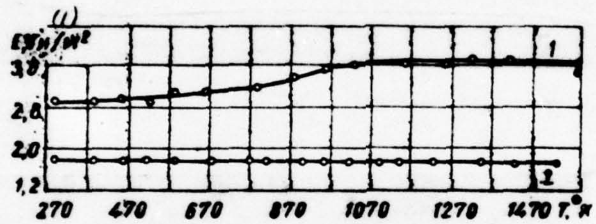

Fig. 71. Temperature dependences of Young's modulus of aluminosilicate refractory: 1 - aluminosilicate refractory without additions of nitride of boron; 2 - aluminosilicate refractory containing 40o/o of nitride of boron.

Key: (1). E, GN/m².

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We have determined the moduli of elasticity of glass No 1 system SiO_2-Al_2O_3-MgO-CaO-TiO_2 and Pyroceram on its basis at the normal and

elevated temperatures. Glass for specimen/samples is cooked in periodic tank furnace in capacitance 1.5 t and then it is produced by hand in the form of the molding/bars with a diameter of 8 mm. These molding/bars are cut to the specimen/samples with a length of 90 mm. Crystallization is conducted in furnace SKB-7074 [51].

Young's modulus and displacement are determined from the measurements of the resonance frequencies of the flexural and torsional oscillations with setting up UP-6. Was accepted the following measurement procedure. Specimen/sample places into the high-temperature camera/chamber the setting up UP-6 and at room temperature measure the moduli of elasticity. Then furnace slowly heats to temperature of 370°K, and resonance frequency measures only after holding at this temperature during 30 min. The duration of holding to measurement at other temperatures comprises: with 470°K - 20 min, 570-670°K - 10 min, 770-870°K - 5-10 min, 970-1070°K - 5 min.

The determination of the moduli of elasticity at room temperature carries out in 10 specimen/samples. Were obtained the following average values of elasticity characteristics: for glass - $E=89$ GN/m², $G=36.3$ GN/m², $\mu=0.2$; for Pyroceram - $E=101$ GN/m², $G=42$ GN/m², $\mu=0.2$.

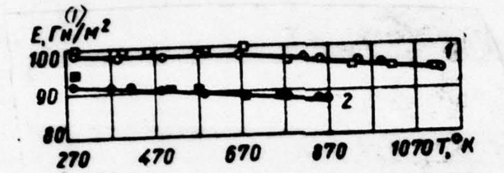

Fig. 72. The temperature dependence of Young's modulus of Pyroceram (1) and of glass (2).

Key: (1). E, GN/m².
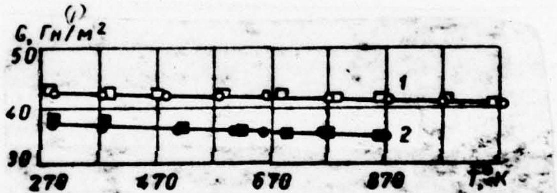
Fig. 73. Temperature dependence of modulus of shear of Pyroceram (1) and of glass (2).

Key: (1). GN/m².

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Deviations of the separate values of E and G from average values compose 4-8o/o, which apparently, is connected with the great effect of thermal history on the structure of glass and especially Pyroceram.

The values of elastic constants during heating were measured up to temperature of 870°K for glass and by 1070°K for Pyroceram. At the higher temperatures of measurement, we cannct be carried out, in view cf strong oscillation damping. The results cf measurements, carried out in two specimen/samples of each material, are given to Fig. 72 and 73. As is evident, both of module/moduli with an increase in the temperature change insignificantly - approximately by 5o/o in the temperature range being investigated.

4. Metal-glass materials.

Together with the development of new vitreo-crystalline materials, are conducted considerable works on the creation of metal-glass materials. Such materials have high corrosion resistance, since in them the open pores are filled by the glass, which eliminates the access of aggressive reagents into the depth of material, and also good wear resistance, since there is a combination cf soft metallic matrix/die with sclid glass connection/inclusions.

Work [6] gives the results of the investigations of strength for breakage and bend, and also the impact toughness of the sintered ferro-glass materials with the content of glass 0.5; 1; 3; 5; 7 and

12 mas.o/o. The authors [6] arrived at the conclusion that the strength of metal-glass materials is determined by the strength of metal frame. The glass, introduced into charge, virtually without strengthening material, contributes to the development of the processes of the shrinkage of metal frame and thereby to an increase in the mechanical properties of metal-glass materials. However, it should be noted that by these authors were investigated such mechanical properties whose decisive importance has plasticity of material, determined first of all by metal frame. In connection with this it is interesting to explain a question concerning the effect of the content of glass on elasticity characteristics of metal-glass material, whose effect by brittle component can be considerable.

We will study the effect of the content of glass on elasticity characteristics of ferro-glass materials with the content of glass from 30 to 12 mas.o/o. Specimen/samples are manufactured in the Institute of the problems of the science of materials of the AS USSR from materials on the basis of the restored/reduced iron powder PZh-2M (GOST [All-union State Standard] 9849-61), containing 0; 3; 5; 12 mas.o/o of glass of brand VVS. Powdered glass is obtained by the grinding of glass-combat in ball mill.

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During the manufacture cf the charge of component, accurately they weigh and thoroughly they mix, and into mixture are introduced C.5-1.0 mas.o/o of machine cil. Specimen/samples are pressed on a 63-ton hydraulic press in rate 30 mm/min and are sintered in furnace G-30 in dried through silica gel hydrogen during 2 h. The sintered blanks are worked on the machine tcols. Ready specimen/samples take the form of the rods with a diameter of 7-8 mm and with a length of 90 mm.

The measurement of Young's modulus and displacement is produced in the rescnance frequencies of the transverse and tcrsional oscillations of specimen/sample with setting up UP-4. Elastic modulus and shear modulus was determined in two-three specimen/samples of one composition at rccm temperature. Measurement data of elasticity characteristics cf the specimen/samples of different compositions are given to Fig. 74.

Specimen/samples made cf pure iron (porosity 7.5o/o) had an elastic modulus 162-183 GN/m², and modulus cf shear 65.5 GN/m², which corresponds to those given in [3] cn this material. With an increase in the content of glass cf the value of Young's modulus and displacement, they are decreased in specimen/samples with the content cf glass 12 mas.o/o E=116 GN/m² and G=45 GN/m². This testifies to the considerable effect of glass on elasticity characteristics of

ferro-glass materials, since glass - material with the low modulus of elasticity.

It is interesting to determine also the effect of an increase in the temperature on elasticity characteristics of ferro-glass materials, since the components of these materials in different ways change their elasticity characteristics during heating. The moduli of elasticity of iron considerably are decreased with temperature, and glass remain almost without change.

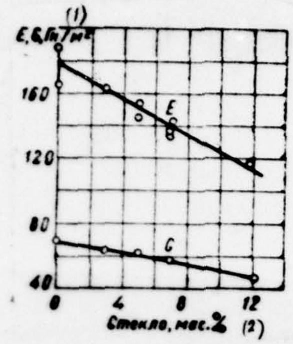

Fig. 74. Dependence of Young's modulus and modulus of shear of ferro-glass materials on the content in them of glass.

Key: (1). E, GN/m². (2). Glass, mas.%.

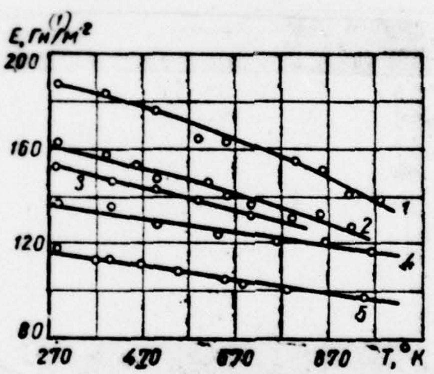

Fig. 75. Temperature dependences of Young's modulus of ferro-glass materials:

1 — Fe; 2 — Fe+3% стекла; 3 — Fe+5% стекла; 4 — Fe+7% стекла; 5 — Fe+12% стекла.

Key: (1). E, GN/m². (2). glass.

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The measurements of the moduli of elasticity of ferro-glass materials in the range of temperatures from room to 970°K are conducted with setting up UP-6. The obtained temperature dependences of the elastic modulus are represented in Fig. 75.

An increase in the content of glass has sufficiently essential effect on a change in Young's modulus with a change in the temperature. So, if in pure iron Young's modulus in the temperature range indicated is decreased by 47 GN/m², then in the ferro-glass material with a content of 12 mas.o/o of glass Young's modulus is decreased by 19.6 GN/m². The obtained temperature dependences of shear mo[illegible] are represented in Fig. 76, from which it is evident that glas[illegible] effect also on the temperature dependence of shear modulus, bu[illegible]s effect is less considerably.

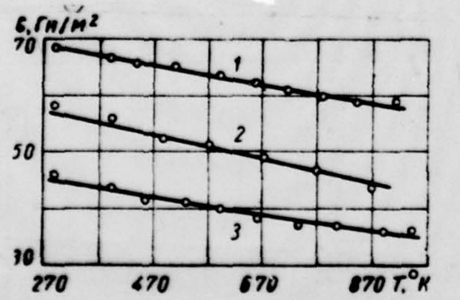

Fig. 76. The temperature dependences of the modulus of shear of ferro-glass materials: 1 - Fe; 2 - Fe + 7% glass; 3 - Fe + 12% glass.
Key: (1). ~~G~~ H N/m^2.

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